THE IONIC HYDROGENATION OF DIACETYLTALATISAMINE

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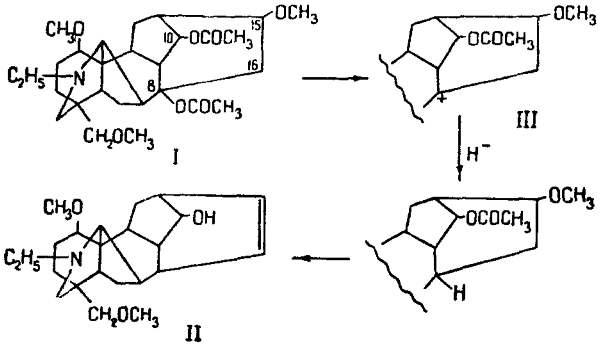
Studying the pyrolysis of diacetyltalatisamine (I) $C_{28}H_{43}NO_7$ in dependence on the conditions of the reaction, we heated it in glycerol at 210-220°C for 2 h. Two products were formed, one of which, $C_{23}H_{35}NO_3$ (II), was isolated in the pure state. This compound has two methoxyls (δ 3.24 and 3.20 ppm; three-proton singlets) and an N-ethyl group (three-proton triplet; 0.97 ppm) and lacks acetyl groups. In the region of olefinic protons of the NMR spectrum at 5.40-5.90 ppm there is a two-proton multiplet. The product contains only one hydroxy group, as was shown by the preparation of a monoacetyl derivative. The fact that this is located at C-10 is confirmed by the presence in the NMR spectrum at 3.96 ppm of a poorly resolved one-proton triplet with $J \approx 4.5$ Hz [1].

Thus, on considering the empirical formula and functional composition of the substance, it may be considered that pyrolysis is accompanied by the elimination of a molecule of methanol, the saponification of the ester group at C-10, and the hydrogenolysis of the acetoxy group at C-8. The similarity of the signals of the olefinic protons in the NMR spectra of the reaction product and of isopyroacetyltalatisamine [1], the mobility of the methoxy group at C-15 [2], and the presence in the mass spectrum of II of a strong $M - 31$ peak (retention of the methoxy group at C-1) [3] shows the presence of a double bond between C-15 and C-16. The hydrogenolysis of an acetoxy group under the conditions of the reaction could apparently take place only as the result of ionic hydrogenation [4]. The carbonium ion formed (III) adds a hydride ion, the donor of which is a glycerol molecule (apparently the proton geminal to the secondary hydroxy group).



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