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Reaction of (20 S)-3 β -Acetoxybisnorcho1-5-enic Acid with Lead Tetraacetate; Structure of Pregnane Derivatives and a Dimer*

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The reaction of lead tetraacetate with carboxylic acids is known to effect oxidative decarboxylation. As a part of our studies on steroid chemistry, we carried out the reaction of 3β -acetoxybisnorchol-5-enic acid (1) with lead tetraacetate, and the results are reported herein.

1 was treated with lead tetraacetate and the products were examined by thin-layer

$$CH_3$$
 CH_3
 CH_3
 CH_3
 CH_3
 AcO
 AcO

Chart 1. Reaction Products of 1 with Lead Tetraacetate

^{*} 本報は Chem. Pharm. Bull., 30, 822 (1982) に報告.

chromatography. Four spots were obtained, and the corresponding substances, 2a, b, c, 3a, b, 4 and 5, were separated by silica gel column chromatography. Their structures were determined as shown in Chart 1.

Reduction of 5 with LiAiH₄ in tetrahydrofuran was then attempted and its reaction products were identified as 3β ,20 α -dihydroxypregn-5-ene (9a) and 3β ,22-dihydroxybisnor-chol-5-ene (11) by GC-MS, mixed mp determination and PMR spectroscopy (Chart 2).

Chart 2.

In view of these results and PMR and MS analyses, the structure of 5 was considered to be $3'\beta$ -acetoxypregn-5'-en-20' α -yl 3β -acetoxybisnorchol-5-enate with the ester linkage at positions 22 and 20'.

Further, the reactions were examined under various conditions and the reaction mechanisms are discussed.