aryl magnesium bromides using the previously published method and were recrystallised from ethanol.

The syntheses of the 4-phenyl and 4-methoxyphenyl derivatives have already been reported. The 4-propyl derivative: reaction time 15 min. M. p. 45.4–46.8°C. (Found: C 68.65; H 6.93. Calc. for C_{14}H_{13}O: C 68.69; H 6.92.)

The 4-isopropyl derivative: reaction time 15 min. M. p. 81.5–82.5°C. (Found: C 68.67; H 7.00. Calc. for C_{15}H_{15}O: C 68.69; H 6.92.)

The 4-o-tolyl derivative: reaction time 15 min. M. p. 145.5–146.5°C. (Found: C 73.51; H 5.80. Calc. for C_{16}H_{16}O: C 73.53; H 5.85.)

The 4-mesyl derivative: reaction time 20 min. M. p. 123.5–125.5°C. (Found: C 74.32; H 6.53. Calc. for C_{17}H_{17}O: C 74.52; H 6.66.)

The 4-o-t-butylphenyl derivative: the ratio of ester to Grignard reagent was increased to 1:5; reaction time 48 h. M. p. 106–107°C. (Found: C 76.21; H 6.79. Calc. for C_{25}H_{35}O: C 74.98; H 6.86.)

The 4-methyl derivative: reaction time 15 min. The compound did not crystallise. After careful evaporation of the solvent, the remaining oil gave an excellent NMR spectrum that showed that the reaction product was ethyl 4-methyl-3,4-dihydro-3-coumarin-oxycarboxylate.

The unsubstituted ethyl 3,4-dihydro-3-coumarinoxycarboxylate was prepared by esterification of 3,4-dihydro-3-coumarincarboxylic acid in ethanol in the presence of dry hydrogen chloride. The reaction product did not crystallise but after careful evaporation of the solvent gave an excellent NMR spectrum that showed that it was ethyl 3,4-dihydro-3-coumarin-oxycarboxylate.

3,4-Dihydro-3-coumarin-carboxylic acid was prepared from 3-coumarin-carboxylic acid by reduction with sodium amalgam according to the method used in the synthesis of N-methyl-3,4-dihydroxyphenylalanine. The melting point, 141–142°C, agrees with the value given in the literature.

5 α,8 α-Peroxyergosteryl Divaricinate from Haematoma ventosum

TORGER BRUUN and ANNE-MARGRETHE MOTZFELDT

Institutt for organisk kjemi, Universitetet i Trondheim, Norges tekniske høgskole, N-7034 Trondheim-NTH, Norway

The crystalline material from Haematoma ventosum (L.) Mass. (for details, see Experimental) gave a mass spectrum which exhibited two important fragmentations. One corresponded to the elimination of divaricatine acid, to give a fragment of composition as ergosterol peroxide which had lost one molecule of water. The other fragmentation corresponded to a further loss of O₂. The observed light absorption of the isolated material, cf. Table 1, was very similar to that of methyl divaricinate.

![Diagram](image)


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Table 1. UV light absorption of isolated and synthetic peroxyroergosteryl divaricinate and of methyl divaricinate.

<table>
<thead>
<tr>
<th>Peroxyroergosteryl divaricinate</th>
<th>Methyl divaricinate</th>
</tr>
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<tbody>
<tr>
<td><strong>Isolated Max.</strong></td>
<td><strong>Synthetic Max.</strong></td>
</tr>
<tr>
<td>ε 302</td>
<td>ε 301</td>
</tr>
<tr>
<td>ε 264</td>
<td>ε 263</td>
</tr>
</tbody>
</table>

mass spectra on an AEI MS902 instrument, and 1H NMR spectra on a Varian A-60A spectrometer.

The material (4.48 kg) was collected in the Oppdal region some 140 km south of Trondheim. It was extracted for 24 h with ether in a glass Soxhlet apparatus. The neutral part (20.3 g) of the material was obtained by extracting the acidic substances with alkali. It was chromatographed on alumina (600 g). The material eluted with ether and ether-methanol 9:1 (3.7 g) was rechromatographed on silica gel, 400 g, retention volume 700 ml. After 500 mg had been eluted with benzene (700 ml) and benzene-ether 99:1 (2100 ml), 162 mg were eluted with the first 700 ml of benzene-ether 49:1. It slowly deposited crystals, which, recrystallised from acetone, melted at 171 – 172 °C, [α]D +1° (c 1.00, 1 dm tube, CHCl₃). M+ at m/e 620 (11 %), found 620.4073, calculated for C₃₅H₅₅O₃ 620.4077; m/e 410 (10 %), C₃₃H₄₁O₂; m/e 378 (100 %), C₃₃H₄₁.

The IR spectrum had bands at 3600 – 3000 cm⁻¹ (OH) and at 1643 cm⁻¹ (C=O). UV spectrum, see Table 1. The 1H NMR spectrum had a singlet at δ 3.80 (OCH₃) and a multiplet at 5.23 (olefinic proton) as the two most interesting features.

Ergosteryl peroxoic acid was prepared according to Windaus and Brunken,⁴ m.p. 176 – 177 °C, reported 178 °C.

Divaricatinic acid chloride was prepared with oxalyl chloride according to Adams and Ulich.⁵ Divaricatinic acid (250 mg) in dry benzene (10 ml) was cooled in ice-water and oxalyl chloride (1.5 ml) was added. After 40 min at room temperature benzene and excess oxalyl chloride were removed in a vacuum whilst nitrogen was passed through.

Peroxyroergosteryl divaricinate. Peroxyergosterol (0.5 g) in dry benzene (10 ml) and redistilled dry pyridine (5 ml) was added dropwise to crude divaricatinic acid chloride. After standing for 18 h the mixture was heated for 1 h on the water bath. The cooled mixture was diluted with ether and extracted with 2 N sulfuric acid, sodium carbonate and water. The residue (0.66 g) was chromatographed on silica gel (60 g). Elution with benzene-ether 99:1 gave a substance which was crystallised from acetone to give long, colourless needles (30 mg), m.p. 169 – 170 °C, [α]D +8° (c 2.00, 1 dm tube, CHCl₃).

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Correction to

"Tobacco Chemistry. 22"*

JOSEPH R. HLUBUCEK, ARNE J. AASEN, SVEN-OLOF ALMQVIST and CURT R. ENZELL

Research Department, Swedish Tobacco Co., S-104 62 Stockholm 17, Sweden

The configurational designation of one of the title compounds, (1'S,6'S)−4-(2',2',6'-trimethyl-6'-vinylcyclohexyl)-2-butaneone, is incorrect and its name should read (1'S,6'R)−4-(2',2',6'-trimethyl-6'-vinylcyclohexyl)-2-butaneone. The structure and stereochemistry of the compound, and the formula given, corresponding to the latter name, are reproduced correctly.

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