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Publication Date

1951-12-07

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Contract No. W-7405-eng-48

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December 7, 1951

Berkeley, California

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ABSTRACT

December 7, 1951

Cholesteryl laurate, m.p. 78-78.5°, has been prepared from cholesterol and lauroyl chloride. This product represents a hitherto unreported isomorphous modification of the ester.

(*) The work described in this paper was sponsored by the U. S. Atomic Energy Commission.

For publication in the Journal of the American Chemical Society.

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Several isomorphous modifications of cholesteryl laurate have been reported. Page and Rudy¹ prepared the ester, m.p. 91° $[\alpha]_D -27.6^\circ$ by heating cholesterol and lauric acid in a current of carbon dioxide; Jaeger² used the same method to obtain an ester m.p. 100°; Cataline and co-workers³ heated the sterol and acid in benzene in the presence of benzene sulfonic acid to obtain this compound, m.p. 92° and Abderhalden and Kautzsch⁴ heated a chloroform solution of cholesterol and lauroyl chloride to obtain a product sintering at 78°, m.p. 110°; $[\alpha]_D -31.3^\circ$. In our hands, heating a pyridine solution of the sterol and acid chloride⁵ yielded an ester, m.p. 78-78.5°; $[\alpha]_D^{21} -30.3^\circ$. Two other preparations of this ester following the methods of Page¹ and of Cataline³ gave material melting at 75-76° and 74-75° respectively. Crystallizations from acetone, ethanol, or chloroform-methanol did not alter the melting point in any case. A sample of our material maintained at 75° for 100 hours melted over the range 75-85°. Another sample, maintained at 80° for 100 hours melted, after solidification, over the range 60-78°.

Our preparation would appear to be a hitherto unreported isomorphous modification of cholesteryl laurate.

EXPERIMENTAL⁶

Starting materials: - Cholesterol (Amerchol), m.p. 141-143°; Lauric Acid (Eastman Kodak), m.p. 37°. The acid chloride gave quantitative yields of the amide, m.p. 99° (lit.⁷ 98°) and anilide, m.p. 75° (lit.⁷ 76°) when treated with ammonium hydroxide and aniline respectively.

Cholesteryl laurate: - A solution of 28 g. of cholesterol and 17.5 g. of lauroyl chloride in 25 cc. of pyridine was heated to boiling, and, after one minute, allowed to cool. The brown solid obtained on cooling was dissolved in ether, washed free of excess pyridine and acid and the ether dried over anhydrous sodium sulfate. Distillation of the ether left a tan solid, m.p. 73-76°. Three crystallizations from chloroform-methanol yielded 33 g. (80%) of white needles, m.p. 78-78.5° $[\alpha]_D^{21} -30.3^{\circ}_m$ (CHCl₃).

Anal:⁸ Calcd. for C₃₉H₆₈O₂: C, 82.33; H, 12.05. Found: C, 82.21; H, 11.99.

Cholesteryl laurate¹: - Lauric acid (4 g.) and cholesterol (2 g.) were heated in a stream of carbon dioxide at 200° for 3 hours. After removal of excess acid the ester was obtained as white needles from chloroform-methanol, m.p. 75-76° $[\alpha]_D^{19} -30.2^{\circ}$ (CHCl₃). Mixed melting point with first preparation, 75-76°.

Cholesteryl laurate³: - A solution of 5.8 g. of cholesterol, 3 g. of lauric acid and 0.13 g. of p-toluenesulfonic acid in 75 cc. of benzene

was allowed to reflux, under constant water take-off, for 3 hours. After removal of all acidic material, the ester was crystallized from chloroform-methanol, M.P. 74-75°. $[\alpha]_D^{21} -28.8^\circ$ (CHCl₃). Mixed melting point with the initial preparation, 74-75°; mixed melting point with the second preparation, 75-76°.

SUMMARY

Cholesteryl laurate, m.p. 78-78.5°, has been prepared from cholesterol and lauroyl chloride. This product represents a hitherto unreported isomorphous modification of the ester.

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