Title
CHOLESTERYL LAURATE

Authors
Kritchevsky, David
Anderson, Margaret E.

Publication Date
1951-12-07

UCRL-

UNIVERSITY OF CALIFORNIA — BERKELEY

RADIATION LABORATORY

DISCLAIMER

UCRL-1600
Unclassified-Chemistry Distribution

UNIVERSITY OF CALIFORNIA

Radiation Laboratory

Contract No. W-7405-eng-48

CHOLESTERYL LAURATE

David Kritchevsky and Margaret E. Anderson

December 7, 1951

Berkeley, California

CHOLESTERYL LAURATE

David Kritchevsky and Margaret E. Anderson

Radiation Laboratory and Department of Chemistry

University of California, Berkeley *

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 isomorphic 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.

CHOLESTERYL LAURATE

David Kritchevsky and Margaret E. Anderson

Radiation Laboratory and Department of Chemistry

University of California, Berkeley *

Several isomorphic modifications of cholesteryl laurate have been reported. Page and Rudy[1] prepared the ester, m.p. 91° $[\alpha]_D$ -27.6° by heating cholesterol and lauric acid in a current of carbon dioxide; Jaeger[2] used the same method to obtain an ester m.p. 100°; Cataline and co-workers[3] heated the sterol and acid in benzene in the presence of benzene sulfonic acid to obtain this compound, m.p. 92° and Abderhalden and Kautzsch[4] heated a chloroform solution of cholesterol and lauroyl chloride to obtain a product sintering at 78°, m.p. 110°; $[\alpha]_D$ -31.3°. In our hands, heating a pyridine solution of the sterol and acid chloride[5] yielded an ester, m.p. 78-78.5°; $[\alpha]_D^{21}$ -30.3°. Two other preparations of this ester following the methods of Page[1] and of Cataline[3] 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 isomorphic modification of cholesteryl laurate.

EXPERIMENTAL[6]

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°m ($CHCl_3$).

Anal:[8] Calcd. for $C_{39}H_{68}O_2$: C, 82.33; H, 12.05. Found: C, 82.21; H, 11.99.

Cholesteryl laurate[1]: - 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° ($CHCl_3$). Mixed melting point with first preparation, 75-76°.

Cholesteryl laurate[3]: - 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 meterial, the ester was crystallized from chloroform-methanol, M.P. 74-75°. $[\alpha]_D^{21}$ -28.8° ($CHCl_3$). 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 higherto unreported isomorphic modification of the ester.

REFERENCES

1. I. H. Page and H. Rudy, Biochem. Z., 220, 304 (1930).
2. F. M. Jaeger, Rec. trav. chim., 25, 334 (1906) quoted by Page and Rudy.
3. E. L. Cataline, L. Worrell, S. F. Jeffries, and S. A. Aronson, J. Am. Pharm. Assoc., 33, 107 (1944).
4. E. Abderhalden and K. Kautzsch, Z. physiol. Chem., 65, 69 (1910).
5. D. Kritchevsky, J. Am. Chem. Soc., 65, 480 (1943).
6. All melting points corrected.
7. R. L. Shriner and R. C. Fuson, "Identification of Organic Compounds," John Wiley and Sons, Inc., New York, 1948. p. 223.
8. Analysis by Mr. V. Tashinian of the microanalytical laboratory, University of California, Berkeley.