

Note on the Preparation of Margaric Acid

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Skraub and Schwamberger¹ described a synthesis of margaric acid by the Barbier-Wieland degradation of methyl stearate. By this method the oxidation of 1,1-diphenyloctadecene gave crude margaric acid in 56% yield. On crystallisation a product of m. p. 60—61° was obtained.

By changing the rather tedious method of isolation of the reaction product we obtained margaric acid m. p. 58—60° in 68% yield, which after one recrystallisation from methanol gave pure acid, m. p. 60—61.5°.

EXPERIMENTAL

Starting from ethyl stearate 1,1-diphenyloctadecene was prepared in 75% yield¹.

Three hundred grams (0,744 mole) of 1,1-diphenyloctadecene were dissolved in 3 l. of glacial acetic acid, and oxidized by gradual addition with stirring of a solution of chromium trioxide (156 g.) in water (180 ml.) at 60—70°. The excess of acetic acid was removed by distillation in vacuo, and the green syrupy residue treated on the water bath 1 to 1,5 hours with 3 l. of 10% sulfuric acid. The mixture was transferred into a beaker and cooled by running water. The lower, sulfuric acid layer, was drawn off, and the crude, solid product washed by suspending it 2 to 3 times in water, and finally pressed dry on a Büchner funnel.

The greenish colored product was dissolved in 1 l. of warm acetone, and made alkaline by addition of 160 ml. of 5 N methanolic potassium hydroxide, cooled, and the precipitated potassium margarate filtered and washed with acetone and finally with ether. The washings were collected, evaporated to dryness, and the residual oil subjected to a second oxidation with $\frac{1}{3}$ of the previously mentioned amount of chromium trioxide. The crude acid was converted to its potassium salt in the same manner as described above. Thus, in total, 184 g. of potassium margarate were obtained.

The salt was decomposed with aqueous hydrochloric acid, and the precipitated crude margaric acid (153 g., i. e. 76% m. p. 56—58°) filtered and dried. For purification the acid was distilled in vacuo. B. p. 186—188°/0,5—1 mm. Yield 136 g. or 68%, m. p. 58—60°. One recrystallisation from methanol yielded 124 g. of pure margaric acid, m. p. 60—61,5°. (m. p. 60°, 62°)^{2, 3}.

REFERENCES

1. S. Skraup and E. Schwamberger, *Ann.* 462 (1928) 135.
2. Shriner, Fulton and Burks, *J. Am. Chem. Soc.* 55 (1933) 1494.
3. Francis, Piper and Malkin, *Proc. Roy. Soc. (London) A.* 128 (1930) 214.

IZVOD**Bilješka o pripremi margarinke kiseline***D. Sunko i P. Alaupović*

Modifikacijom Skraup i Schwamberger-ove metode pripreme margarinke kiseline Barbier-Wieland-ovom odgradnjom stearinske kiseline¹, postignuto je iskorištenje na čistoj kiselini od 68% računano na 1,1-difeniloktadecen, dok je prema originalnom postupku iskorištenje na sirovoj, nekristaliziranoj kiselini 56%.

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