

literature as 285° is in error due to the presence of lower glycerides. A boiling point of $305\text{--}310^{\circ}$ is established as more nearly accurate.

The butyric acid used in these determinations was guaranteed by the maker to be normal butyric acid of the highest purity. From a 0.9kg. lot a fraction of 484 cc. was obtained having a boiling point of $162\text{--}163^{\circ}$ (*n*-butyric acid, b. p., 162.5°). Chemically pure glycerol was used which was further dehydrated by heating under reduced pressure.

Color, Odor and Taste.—The purified tributyrin is colorless. It has a characteristic, though not unpleasant odor. It possesses a markedly bitter taste, noticeable especially in the palate area of the throat. It was thought that this taste was probably due to decomposition products, but repeated distillation under reduced pressure did not remove the substance that caused it.

Specific Gravity.—The specific gravity at 21° of the tributyrin purified by vacuum distillation was found by pycnometer measurement to be 1.027.

Freezing Point.—Freezing-point determination of the purified tributyrin was made by means of carbon dioxide snow and ether, using a standardized toluene thermometer. The butyrin began to thicken at about -35° , and at -75° , the lowest temperature obtained, it was so viscous that the thermometer could scarcely be withdrawn; yet it had not solidified. This behavior may be due in part to traces of the lower glyceride, though primarily to the nature of the substance itself.

Summary

The rate of esterification of glycerol and butyric acid was determined. The effect of mass in forcing the esterification more nearly to completion and in increasing the yield of the triglyceride was established.

Purified tributyrin was isolated and its properties established. It is a colorless liquid with characteristic odor and bitter taste; d^{21} , 1.027; b. p., about 190° (15 mm.), or $305\text{--}310^{\circ}$ (atmos.). At lower temperatures it thickens to a viscous mass which does not solidify at -75° .

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NOTE

Rapid Preparation of Cetyl Alcohol.—No simple, rapid method of obtaining cetyl alcohol from spermaceti has been published. Beilstein¹ gives a very tedious preparation, involving saponification with potassium hydroxide in ethyl alcohol, precipitation of the cetyl alcohol and calcium palmitate with calcium chloride, filtration, drying and extraction with alcohol. Axelrad² gives also an unsatisfactory method with the added

¹ Beilstein, "Handbuch der organischen Chemie," Springer, Berlin, 1918, 4th ed., vol. I, p. 429.

² Axelrad, *J. Ind. Eng. Chem.*, 9, 1123 (1917).

possibility of the product being mixed with other substances produced by a distillation at atmospheric pressure of a mixture of cetyl alcohol and calcium palmitate.

Contrary to statements in the literature, spermaceti, although a wax, can be saponified with extreme ease and rapidity and the cetyl alcohol extracted from solution by shaking with petroleum ether. It is necessary, however, to control the proportions of water, ethyl alcohol and saponification products, since thick, semi-solid gels, or semi-permanent emulsions or suds are easily produced. By the method given below, several hundred grams of crude cetyl alcohol can be prepared in a few hours with ordinary apparatus, or 50-100 g. in less than two hours. The principal impurity is probably octadecyl alcohol.³

A mixture of 100 cc. of 95% alcohol and 100 g. of spermaceti in a flask is heated on the steam-bath until the wax melts. A solution of 12 g. of potassium hydroxide in 10-12 cc. of water is added slowly (to prevent boiling off of alcohol by the heat of reaction) and the mixture is stirred or shaken. The two layers mix in one-half to two minutes. The solution is warmed and agitated for 15 minutes longer. It is then transferred to a 1000cc. separatory funnel, the flask washed out with 75 cc. of alcohol and 200 cc. of water, and the washings are added to the funnel. While the solution is still slightly warm, 400 cc. of petroleum ether is added and the mixture shaken vigorously. If a gel or a slowly-breaking emulsion forms, 10 cc. of alcohol at a time is added and the mixture reshaken until, on standing, the layers separate readily. The aqueous layer is drawn off and extracted four to eight times with 200cc. portions of petroleum ether. The solvent is evaporated from the combined extracts by distillation, but the last portions can be removed by blowing a current of air on the surface of the liquid while on a steam-bath, or in a vacuum. Typical extraction results are given in Table I.

TABLE I

Extract No.	1	2	3	4	5
Wt., g.	22.6	9.8	5.6	3.8	3.0
Total yield, calcd.: 51.4. Found: 44.8. M. p., 45-46°.					

Nearly all can be recovered by three more extractions. The proportions of wax, alcohol and water indicated above cannot be much modified if emulsions or gels are to be avoided. The chief advantage of the method is its rapidity, and the chief disadvantage is the large quantity of solvent required. Ether cannot be used for extraction because of the large proportion of alcohol present. Ordinary crude alcohol can be used in saponification, since the colored resinous impurities produced by the potassium hydroxide are insoluble in petroleum ether and a nearly white product is

³ Kraft, *Ber.*, 17, 1628 (1884).

obtained. To prepare larger quantities, successive portions of saponification mixture may be extracted with the dil. petroleum ether extracts from the earlier extractions, to economize in the use of solvent. For purification, the alcohol is best converted to the acetate (by passing dry hydrogen chloride into a warm solution in 2 to 3 molecular equivalents of acetic acid), and this fractionated at reduced pressure, yielding cetyl acetate; m. p., 22.7°; b. p., 184° (5 mm.) (corr.). Partial purification is possible by several recrystallizations of the spermaceti from acetone before saponification. The alcohol cannot be purified by recrystallization except with considerable loss.

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NEW BOOKS

The Effects of Ions in Colloidal Systems. By Dr. LEONOR MICHAELIS, University of Berlin. At present, Professor of Biochemistry at the Aichi Medical University in Nagoya, Japan. Williams and Wilkins Company, Baltimore, Maryland, 1925. 108 pp. 7 figs. 13 × 19 cm. Price \$2.50.

This little book is based on a series of lectures given by the author last year in this country. It deals with the colloid chemistry of aqueous solutions, largely from the viewpoint of the adsorption of ions. Some of the topics considered are the origin of electric double layers, the adsorbent properties of charcoal, the flocculation of colloids by electrolytes, the Donnan equilibrium and the lyotropic effects of ions. The discussion is based on experiments taken, for the most part, from the author's own work.

One source of confusion in colloid chemistry has been the danger of generalizing from experiments made with material whose nature was too little understood. Thus, a difference of opinion between Michaelis and Bayliss seems to have been due to the latter's failure to recognize the presence of calcium in filter paper. Some of Michaelis' own generalizations about adsorption by charcoal have had to be qualified in the light of recent American work with pure-sugar charcoal instead of blood charcoal. The classification of agar as electrically indifferent is not in accord with recent work indicating that it behaves as an ionizable calcium salt of a carbohydrate-sulfuric acid.

Apparently it was impossible for such productive workers as Professor Michaelis and Jacques Loeb to keep fully posted on each other's writings. Thus Loeb's suggestion concerning the possible capillary activity of hydroxyl ion turns out to have been made by Michaelis several years before. In criticizing Loeb's attempt to identify cataphoretic potentials with mem-