

chloride. The acid aniline salt above described, melting at  $246^{\circ}$  was precipitated.

Calculated for  $C_9H_8O_3 \cdot 3H_2O$ , 19.22 per cent.

Found:  $H_2O$ , 20.36 and 20.00 per cent at  $110^{\circ}$ .

The results are slightly high, but when it is remembered that these crystals contain some hydrobromic acid, which would also be lost at this temperature, they may be considered conclusive.

The above results are published on account of a temporary interruption of the work. It will, however, be again taken up, and I desire to reserve the field of the halogen derivatives of *p*-sulphocinnamic acid for some time longer.

My hearty thanks are due to my assistant, Mr. L. A. Salinger, who did most of the manipulative work, and conducted most of the analyses recorded in this paper.

MASSACHUSETTS INSTITUTE OF TECHNOLOGY,  
April 13, 1903.

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## THE ANALYTICAL CONSTANTS AND COMPOSITION OF MYRTLE WAX.

BY WARREN RUFUS SMITH AND FRANK BERTRAM WADE.

Received April 10, 1903.

MYRTLE wax, also known as laurel wax or bayberry tallow, is a fat of minor commercial importance obtained from the fruit of *myrica cerifera*, a shrub common along the North Atlantic sea-coast and in other sections of this country. The statements regarding its composition are somewhat contradictory. The general books of reference state that it is mainly free palmitic acid with about 20 per cent. palmitin. Some state that it also contains lauric acid and others that it contains stearic and myristic acids. The first of these statements is due to Moore,<sup>1</sup> the second to Chevreul.<sup>2</sup>

On the other hand, the works on oil and fat analysis state that myrtle wax is a triglyceride with a small amount of free fatty acid, and give low "acid values" in support of these statements. Benedict<sup>3</sup> states that it is composed mainly of palmitin with some myristin and stearin. Lewkowitsch<sup>4</sup> says that in addition to the

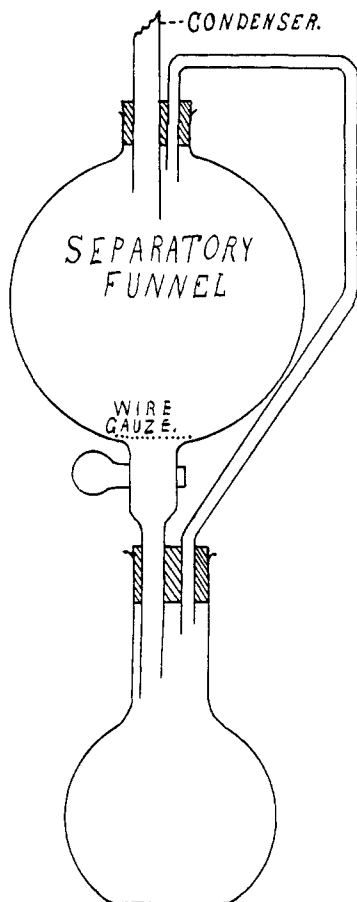
<sup>1</sup> *Am. J. Sci. and Arts*, 2nd series, **33**, 313.

<sup>2</sup> *Compt. rend.*, **20**, 38.

<sup>3</sup> "Analyse der Fette," 3rd ed., p. 535.

<sup>4</sup> "Chemical Analysis of Oils, Fats, and Waxes," p. 542.

above it contains a small amount of oleic acid. Allen,<sup>1</sup> while he does not give the composition of myrtle wax, says that it contains 0.12 per cent of free acid (in terms of palmitic acid). Bayberry



wax has been suggested and used<sup>2</sup> as a convenient source of palmitic acid so that the question of its composition becomes a matter of importance.

The following experiments were undertaken for the purpose of obtaining additional evidence on this question. The material used was obtained by us directly from the berries by extraction with

<sup>1</sup> "Commercial Organic Analysis," 3rd ed., Vol. II, Part 1, p. 169.

<sup>2</sup> Chittenden and Smith: *Am. Chem. J.*, 6, 218.

petroleum ether. The extractor used was devised from ordinary laboratory apparatus and worked so well that we append a sketch of it for the benefit of any one who has occasion to extract quantities of material larger than can be contained in a Soxhlet's extractor of ordinary size.

The following constants were obtained by the methods given in Lewkowitsch's "Chemical Analysis of Oils, Fats and Waxes."

Specific gravity, $\frac{22^\circ}{15.5}$ .....	0.9806
Specific gravity, $\frac{99^\circ}{15.5}$ .....	0.878
Melting-point.....	48° <sup>1</sup>
Solidifying point.....	45°
Saponification value.....	217
Iodine value (Hübl).....	3.9
Reichert-Meissl number.....	0.5
Acid number.....	30.7
Refractive index, 80°.....	1.4363

These figures show the absence of oleic and volatile acids and agree with the assumption that myrtle wax is mainly palmitin with some lower glyceride and a small amount of free acid. By crystallizing four times from petroleum ether pure palmitin was obtained, melting-point, 62.5; saponification value, 209.4; refractive index, 1.4380 at 80°.

Like most fats, myrtle wax shows a change of melting-point and other constants on standing. The berries from which the above wax was obtained were picked in September and the wax was extracted and its constants obtained in November. On extracting another portion in March, a melting-point of 54° was obtained. Wax from berries obtained in August and extracted in March melted at 55.4°. A redetermination in March of the melting-point of the main portion gave 52.5°, a rise of 4.5° in four months. A specimen of wax about four years old melted at 57° and solidified at 58.3°. These rather high figures led us to suspect that stearin might be present but, after repeated crystallization from alcohol, practically pure palmitin was obtained, melting at 61.5° and giving a saponification value of 206.7. The absence of stearic acid was finally shown by the fact that Hehner and Mitchell's method gave no stearic acid even on salting with a crystal of stearic acid and

<sup>1</sup> "Method of Allen," Vol. II, Part I, p. 34.

allowing to stand for a further period of ten hours at 0°. We conclude therefore that myrtle wax contains no stearin and that the high melting-point of the older material is due to the obscure change which takes place in many fats on standing. We hope to study this change in myrtle wax further and determine how the constants, other than melting- and solidifying-points, are affected thereby.

LEWIS INSTITUTE, CHICAGO, ILL.,  
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## THE ANALYSIS OF BRONZES AND BEARING METALS.<sup>1</sup>

BY H. E. WALTERS AND O. I. AFFELDER.

Received April 11, 1903.

In the laboratory with which the writers are connected, many bronzes and bearing metals are analyzed, and it was thought that the methods which are used might prove of interest to the members of this Section.

While the scheme contains much that is old, we wish to call particular attention to the volumetric method for determining lead, as the results obtained by it are as reliable as those obtained by the sulphate method.

### BRONZES.

The analysis of bronzes is carried out as follows:

Weigh 1 gram of the sample (0.5 gram, if the lead is over 15 per cent.) into a No. 2 beaker, cover with a watch-glass, add 10 cc. nitric acid (sp. gr. 1.42) and warm until all is dissolved. When in solution, add 40 cc. hot water and boil five minutes, filter, wash with 2 per cent. nitric acid, burn and weigh as SnO<sub>2</sub>. To the filtrate add 25 cc. strong ammonia and heat to boiling, then add about 5 grams ammonium persulphate and boil from five to ten minutes. Make acid with sulphuric acid, filter and wash with hot water. The lead will remain on the filter as lead peroxide. Transfer the precipitate and filter to the beaker in which precipitation was made, add water and stir well to disintegrate the filter-paper. Dilute to 600-700 cc. with cold water, add about 3 grams potassium iodide and some starch solution. When all the iodide is dissolved, add 10 cc. hydrochloric acid (1:1), stir well and titrate

<sup>1</sup> Read before the Pittsburg Section of the American Chemical Society at the first monthly meeting, March 19, 1903.