

W. T. Carroll

1903



THE RESULTS OF AN EXAMINATION OF NEHALEM WAX.

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The sp. gr. of the wax was determined by means of the Westfall balance. Alcohol and water were mixed in such proportions as would admit of the wax being suspended half way between top and bottom of the liquid, and diverse opinions expressed as to its constitution. Through our papers subsequently the sp. gr. of the liquid taken.

Different men have expressed different opinions, some of these opinions were based alone on physical properties, others on the results of analysis which were evidently very limited. A complete analysis of the substance was made, both at the Smithsonian Institute and at the Geological Survey office in Washington. The results obtained by these examinations identify the substance with bees-wax.

On the coast near the mouth of the Nehalem river have been found pieces of wreckage which are on exhibition in the museum at Portland. An old story makes the deposit of wax the cargo of a wrecked vessel. This cargo of wax was washed upon the sandy shore and buried by the action of the waves and tides. The enormous deposit of wax is at the mouth of the Nehalem river, on the coast of Oregon, south of Astoria. Visits to this locality are attended with great difficulty, hence an eye witness description is impossible here. The quantity of wax deposited in the district has not been estimated; however the Oregonian has stated that seventeen thousand pounds of the wax have been taken out.

In the analysis of the wax it was desirable to run samples of pure bees-wax and Ozokerite in conjunction with the Nehalem. The samples used in the analysis will be taken up separately and the physical and chemical properties given in detail.

SAMPLE #30. This sample was obtained from L.C. Skeels, Eugene, and was supposed to be pure bees-wax. It was a dark yellowish brown, had the characteristic odor and taste of bees-wax, was very soft and plastic and was soluble to quite a degree in alcohol.

The iodine or Hüble values were obtained as follows. Iodine sol-

The sp.gr. 0.965 was determined by means of the Westfall balance. Alcohol and water were mixed, in such proportions as would admit of the wax being suspended half way between top and bottom of the liquid, and subsequently the sp.gr. of the liquid taken.

The melting point, 63°, was determined by a method introduced by two Japanese, which was simply the suspension of a platinum carrier, containing a layer of the wax between thin glass plates; in an air bath which was in turn surrounded by a water bath. This contrivance insured a steady and uniform rise in temperature.

The solidification point was determined by placing a thermometer in the melted wax and noting the temperature at which the wax was completely solidified.

All chemical values were determined in duplicate. The acid value was determined by titrating the sample of wax dissolved in alcohol, with a standard solution of sodium hydroxide and calculating the amount of cerotic acid necessary to react with said sodium hydroxide used. The acid value for this sample was, (a) 22.35; (b) 22.01.

The saponification value was determined as follows. Run in from a burette standard alcoholic potassium hydroxide in excess of the quantity necessary to saponify the wax; effected saponification by attaching a reflux condenser and boiling for one hour or more. Standard acid was then used to titrate back for excess of alkali solution. From the above data, the amount of potassium hydroxide required to affect the saponification of the wax, can be calculated. The values are given in terms of the number of milligrams required to saponify one gram of the wax. The two samples (a) and (b) of this wax were lost by breaking the containers.

The sample is identical with sample #32. The following are the experimental values: Sp.gr. 0.961; M.P. 57.7°; solidification point 61°;

The iodine or Hüble values were obtained as follows. Iodine solution from a burette was run into the sample, dissolved in chloroform, and allowed to stand from two to four hours to insure complete absorption of the iodine by the wax. The samples were then titrated with sodium thiosulphate for excess of iodine and from the above data the amount of iodine absorbed was calculated. The iodine value for (a) lost; (b) 11.82.

SAMPLE #31. The sample was furnished by H.A. Vincent. It was produced in the vicinity of Eugene. This sample of bees-wax was very light yellow, had odor and taste of bees-wax but not as strong as sample #30; was much harder^{than} and not as plastic as sample #30, was quite soluble in alcohol. The following are the experimental values determined: Sp.gr. 0.963; M.P. 62.5°; solidification point 62.5°; acid value, (a) 22.12, (b) 22.79; saponification value (a) 121.90, (b) 74.92; Hüble value (a) 7.04, (b) 5.18.

SAMPLE #32. This sample of Nehalem wax was furnished by Geo. H. Haines and was said to have been broken from a piece containing a bee. This sample like all others of Nehalem wax was covered with a thin layer of dirt and sand. It was a very dark brown varying to a light brown towards the center of the pieces, had a decided odor and taste of bees-wax, was hard and little plastic as sample #31, was quite soluble in alcohol. The following are the determined values: Sp.gr. 0.967; M.P. 57.5°; acid value (a) 11.91, (b) 11.89; saponification value 127.8; iodine value (a) 6.36, (b) 6.34.

SAMPLE #33. This sample was furnished by A.C. Kinsey as a true sample of Nehalem wax. Kinsey is a physician of Astoria who made some slight analysis of the substance and published his conclusions. This sample is identical with sample #32. The following are the experimental values: Sp.gr. 0.961; M.P. 57.7°; solidification point 61°; acid value (a) 11.82, (b) 11.82; saponification value (a) 121.90, (b) 74.92; Hüble value (a) 7.04, (b) 5.18.

SAMPLE #34. This sample of Nehalem wax was furnished through the kindness of Hines of Portland, secretary of the Historical Society. In the Portland museum are several large blocks which appear to have characters engraved on them, and it was from one of these, (a) lost; (b) 11.82.

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acid value (a) 8.76, (b) 8.83; saponification value (a) 114.20, (b) 105.90; iodine value (a) 5.20, (b) 5.14.

SAMPLE #34. This sample of Nehalem wax was furnished through Ray Willoughby, by Himes of Portland, secretary of the Historical Association. In the Portland museum are several large blocks which appear to have characters engraven on them, and it was from one of these, that this sample was taken. The odor, taste, hardness and solubility agreed with sample #32. The color ranged from black on the outside to a golden yellow on the inside. The following results were calculated from experiment: Sp.gr. 0.960; M.P. 58.5°; acid value (a) 10.77, (b) 10.55; saponification value (a) 107.90, (b) 89.48; iodine value (a) 5.70 (b) 5.79.

SAMPLE #35. This sample of Ozokerite which came from New York, was run in conjunction with the others to indicate how dissimilar the results of analysis were from those of bees-wax and also Nehalem wax. It was black, had a very strong odor and taste of petroleum, was very soft and plastic and was practically insoluble in alcohol. The results obtained in the analysis were; sp.gr. 0.934; M.P. 58.7°; acid value (a) 0.00, (b) 0.24; saponification value (a) lost, (b) 13.07; iodine value (a) 0.00, (b) 0.26. These results would probably indicate values which the wax has not; due to the fact that in the black solution an end point was almost impossible, especially with any degree of certainty. The values here would account for but minute quantities of solution.

SAMPLE #36. This sample of Nehalem wax was sent through Cleveland by T.B. Elmore of Tillamook. The wax differed in some properties from the preceding samples. It was almost white, had no decided odor or taste, was very hard and brittle and was quite soluble in alcohol. The following values were determined: M.P. 61.5°; acid value

(a) 9.00, (b) 12.57; saponification value (a) 109.50 (b) no sample; iodine value (a) 4.49, (b) 4.26; The original samples of two of the waxes, samples #36 and #37, were so small that they were not run in duplicate.

SAMPLE #37. This sample of Nehalem wax was furnished by H.S.Lamb of Tillamook. It was found under the roots of a large tree presumably in the region of the deposit. In odor, color, taste hardness and solubility it agreed with sample #36. The results of experiment were as follows: M.P. 56°; acid value (a) 8.95 (b) 8.82; saponification value (a) 107.40, (b) no sample; iodine value (a) 4.63, (b) 4.52.

SAMPLE #38. This sample of Nehalem wax was also furnished by H.S.Lamb. Its color, odor, taste, hardness and solubility are the same as sample #32. The experimental results were: M.P. 59.2°; acid value (a) 11.65, (b) 10.64; saponification value (a) 109.10, (b) lost by breakage; iodine value (a) 5.76, (b) 6.43.

#	M.P.	Sp. gr.	Acid value.	Sapon. value.	Iodine value.
30	68°	0.965	22.35		
			22.01		11.82
31	62.5°	0.963	22.12	121.90	7.03
			22.79	74.92	5.18
32	57.5°	0.967	8.69	127.20	8.32
			8.73	98.06	6.34
33	57.7°	0.961	8.76	114.20	5.20
			8.38	105.90	5.14
34	58.5°	0.960	10.77	107.96	5.70
			10.55	89.48	5.79
35	58.7°	0.934		13.07	0.26
36	61.5		9.00	109.50	4.49
			12.57		4.26
37	56°		8.95	107.40	4.62
			8.82		4.26
38	59.2°		11.65	109.10	5.76
			10.46		6.42

The above schedule is a brief summary of the important results.

Some widely differing percentages results occur between the duplicate samples which may be explained; first, by the difficulty of distinguishing a sharp end point in the colored solutions; secondly, by the necessarily small samples; the average sample being less than four grams; thirdly, by the strength of the solutions and fourthly, by the number of burette readings necessary for each sample.

The most important compounds of which bees-wax is composed are Cerotic acid, Myrissal alcohol, Myricin and some salts of the higher acids. The preparation of Cerotic acid from Nehalem wax and bees-wax also the preparation of Myrissal alcohol from the same were not satisfactory; the chief difficulty being in the identification of the substances obtained. Literature on the properties of these substances gives only the melting points and crystal form. Time would not admit of a purification of the substances obtained to a constant melting point.

After a careful study of the properties of many of the waxes and the analysis of the three waxes; bees-wax, Nehalem wax and Ozokerite, side by side, the only conclusion to be reached at present is that Nehalem wax is not Ozokerite and that it does not give the values of bees-wax but that the values determined are more closely connected with those of bees-wax than any other substance.