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## The Extraction of Waxes and Wax Mixtures by Means of Common Organic Solvents

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TABLE OF CONTENTS

	Page
List of Tables .....	1
Preface .....	2
Introduction .....	3
<b>THE EXTRACTION OF WAXES AND WAX MIXTURES</b>	3
<b>BY MEANS OF COMMON ORGANIC SOLVENTS</b>	5
1 - Outline of Thesis .....	5
2 - Outline of Chapter Details .....	7
3 - Outline of Ford Thesis .....	7
Experimental Apparatus, Procedures and Results .....	9
(1) Apparatus .....	9
(2) Sargent Apparatus .....	9
(3) Apparatus .....	11
(4) Apparatus .....	11
(5) Apparatus .....	13
(6) Apparatus .....	13-19
(7) Apparatus .....	20-26
(8) Apparatus .....	27
(9) Apparatus .....	27
(10) Apparatus .....	28
(11) Apparatus .....	28
(12) Apparatus .....	28
(13) Apparatus .....	28
(14) Apparatus .....	28
(15) Apparatus .....	28
(16) Apparatus .....	28
(17) Apparatus .....	28
(18) Apparatus .....	28
(19) Apparatus .....	28
(20) Apparatus .....	28
(21) Apparatus .....	28
(22) Apparatus .....	28
(23) Apparatus .....	28
(24) Apparatus .....	28
(25) Apparatus .....	28
(26) Apparatus .....	28
(27) Apparatus .....	28
(28) Apparatus .....	28
(29) Apparatus .....	28
(30) Apparatus .....	28
(31) Apparatus .....	28
(32) Apparatus .....	28
(33) Apparatus .....	28
(34) Apparatus .....	28
(35) Apparatus .....	28
(36) Apparatus .....	28
(37) Apparatus .....	28
(38) Apparatus .....	28
(39) Apparatus .....	28
(40) Apparatus .....	28
(41) Apparatus .....	28
(42) Apparatus .....	28
(43) Apparatus .....	28
(44) Apparatus .....	28
(45) Apparatus .....	28
(46) Apparatus .....	28
(47) Apparatus .....	28
(48) Apparatus .....	28
(49) Apparatus .....	28
(50) Apparatus .....	28
(51) Apparatus .....	28
(52) Apparatus .....	28
(53) Apparatus .....	28
(54) Apparatus .....	28
(55) Apparatus .....	28
(56) Apparatus .....	28
(57) Apparatus .....	28
(58) Apparatus .....	28
(59) Apparatus .....	28
(60) Apparatus .....	28
(61) Apparatus .....	28
(62) Apparatus .....	28
(63) Apparatus .....	28
(64) Apparatus .....	28
(65) Apparatus .....	28
(66) Apparatus .....	28
(67) Apparatus .....	28
(68) Apparatus .....	28
(69) Apparatus .....	28
(70) Apparatus .....	28
(71) Apparatus .....	28
(72) Apparatus .....	28
(73) Apparatus .....	28
(74) Apparatus .....	28
(75) Apparatus .....	28
(76) Apparatus .....	28
(77) Apparatus .....	28
(78) Apparatus .....	28
(79) Apparatus .....	28
(80) Apparatus .....	28
(81) Apparatus .....	28
(82) Apparatus .....	28
(83) Apparatus .....	28
(84) Apparatus .....	28
(85) Apparatus .....	28
(86) Apparatus .....	28
(87) Apparatus .....	28
(88) Apparatus .....	28
(89) Apparatus .....	28
(90) Apparatus .....	28
(91) Apparatus .....	28
(92) Apparatus .....	28
(93) Apparatus .....	28
(94) Apparatus .....	28
(95) Apparatus .....	28
(96) Apparatus .....	28
(97) Apparatus .....	28
(98) Apparatus .....	28
(99) Apparatus .....	28
(100) Apparatus .....	28

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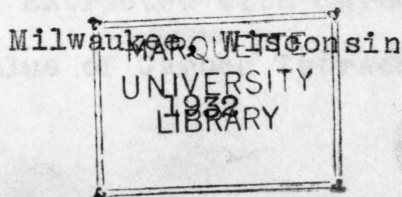


TABLE OF CONTENTS

	Page
List of Tables.....	1
Preface.....	2
Introduction.....	3
A - Outline of Oils, Fats and Waxes.....	3
B - Outline of Severson Thesis.....	5
C - Outline of Hable Thesis.....	6
D - Outline of Spracher Thesis.....	7
E - Outline of Ford Thesis.....	7
Experimental Apparatus, Procedure and Results.....	9
A - Apparatus.....	9
(1) Sargent Apparatus.....	9
(2) Asbestos Chimney.....	11
B - Procedure.....	11
C - Results.....	13
(1) Extraction of seven single commercial waxes with different organic solvents.....	13-19
(2) Extraction of several mixed waxes with dif- ferent organic solvents.....	20-26
The Extraction of Furniture Polish.....	27
A - Procedure.....	27
B - Data.....	28
(1) Percent Extracted with Absolute Alcohol.....	28
(2) Acid Value of Absolute Alcohol Extract.....	28
(3) Saponification Value of Absolute Alcohol Extract	28
(4) Iodine Value of Absolute Alcohol Extract.....	28
(5) Percent Extracted with Carbon Tetrachloride.....	28
(6) Acid Value of Carbon Tetrachloride Extract.....	28

(7) Saponification Value of Carbon Tetrachloride Ext.	28
(8) Iodine Value of Carbon Tetrachloride Extract.....	28
(9) Percent of Unextracted Matter.....	28
(10) Percent extracted with Carbon Tetrachloride.....	28
(11) Acid Value of Carbon Tetrachloride Extract.....	28
(12) Saponification Value of Carbon Tetrachloride Ext.	28
(13) Iodine Value of Carbon Tetrachloride Extract.....	28
(14) Percent Extracted with Absolute Alcohol.....	28
(15) Acid Value of Absolute Alcohol Extract.....	28
(16) Saponification Value of Absolute Alcohol Extract.	28
(17) Iodine Value of Absolute Alcohol Extract.....	28
(18) Percent of Unextracted Material.....	28
The Extraction of Shellac.....	29
A - Procedure.....	29
B - Data.....	32
(1) Percent Extracted with Absolute Alcohol.....	32
(2) Acid Value of Absolute Alcohol Extract.....	32
(3) Saponification Value of Absolute Alcohol Extract.	32
(4) Iodine Value of Absolute Alcohol Extract.....	32
(5) Percent Extracted with Carbon Tetrachloride.....	32
(6) Percent of Unextracted Matter.....	32
(7) Percent Extracted with 95% Alcohol.....	32
(8) Acid Value of 95% Alcohol Extract.....	32
(9) Saponification Value of 95% Alcohol Extract.....	32
(10) Iodine Value of 95% Alcohol Extract.....	32
(11) Percent Extracted with Carbon Tetrachloride.....	32
(12) Percent of Unextracted Matter.....	32

	Page
Experimental Methods used to Characterize Extracted Matter	29
A - Acid Value.....	29
B - Saponification Value.....	29
C - Iodine Value.....	30
Summary.....	33
Bibliography.....	35
5. Extraction of Seven Waxes with Chloroform.....	17
6. Extraction of Seven Waxes with Ether.....	18
7. Extraction of Seven Waxes with Naphtha.....	19
8. Extraction of Seven Mixed Waxes with Carbon Tetra- chloride.....	20
9. Extraction of Three Mixed Waxes with Acetone.....	21
10. Extraction of Three Mixed Waxes with Benzene.....	22
11. Extraction of Three Mixed Waxes with Carbon Disulphide..	23
12. Extraction of Three Mixed Waxes with Chloroform.....	24
13. Extraction of Three Mixed Waxes with Ether.....	25
14. Extraction of Three Mixed Waxes with Naphtha.....	26

LIST OF TABLES

	Page
1. Extraction of Seven Waxes with Acetone.....	13
2. Extraction of Seven Waxes with Benzene.....	14
3. Extraction of Seven Waxes with Carbon Disulphide.....	15
4. Extraction of Seven Waxes with Carbon Tetrachloride.....	16
5. Extraction of Seven Waxes with Chloroform.....	17
6. Extraction of Seven Waxes with Ether.....	18
7. Extraction of Seven Waxes with Naphtha.....	19
8. Extraction of Seven Mixed Waxes with Carbon Tetra- chloride.....	20
9. Extraction of Three Mixed Waxes with Acetone.....	21
10. Extraction of Three Mixed Waxes with Benzene.....	22
11. Extraction of Three Mixed Waxes with Carbon Disulphide..	23
12. Extraction of Three Mixed Waxes with Chloroform.....	24
13. Extraction of Three Mixed Waxes with Ether.....	25
14. Extraction of Three Mixed Waxes with Naphtha.....	26

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## PREFACE

This work was performed with the intention of giving some information of the solubility of the more common waxes, by means of extraction with several commercial solvents. Besides the individual waxes, various mixtures of waxes were also used to determine the effect of the mixtures on the extractability.

Determinations are also made to find the extractable matter in a commercial furniture polish and shellac. The acid, saponification and iodine values of the polish and shellac are also found.

Very little work seems to have been done on the accurate determination of percentage of extractable matter in waxes and mixtures of waxes. For this reason the references made throughout the work concern waxes in general and not their extractability.

## ACKNOWLEDGEMENT

The author expresses his appreciation to Dr. John R. Koch of Marquette University, and Dr. J. Vernon Steinle of S.C. Johnson Wax Company, for their valuable suggestions and help in making this work possible.

(1) Lewkowitsch, J. - Chemical Technology of Oils and Fats, MacMillan and Company, London, 1913, Vol. I, Page 65.

(2) Gowen, T.W. - Wax Craft, Sampson, Low, Marston Company, London, 1903, Pages 13 - 44.

(3) Wright, G.H. - Animal and Vegetable Fixed Oils, Fats, Gums and Waxes - J.B. Lippincott Co., Philadelphia, 1903, Page 10.

## INTRODUCTION

The history, chemistry and classification of the waxes is given very briefly in this work. A number of other papers on waxes have been submitted, and in each of them the above mentioned material has been treated at some length.

A wax is defined by Lewkowitsch<sup>(1)</sup> as an ester formed by the combination of mono- or dihydric alcohols with higher fatty acids.

The history of the waxes, according to Cowan,<sup>(2)</sup> go back very far in history. References are made to wax in many of the legends that have come down through the ages. The Greeks and Romans used wax and knew how to bleach it. In the Middle Ages wooden tables were covered with a thin layer of wax and characters traced in the wax. During the reign of Louis XIV much sculptor work was done in wax. Wax candles were also used to light the homes in earlier times. At the present time there are also numerous uses for waxes.

The following is a classification from Wright,<sup>(3)</sup> of oils, fats and waxes according to their chemical composition.

### Division I - Hydrocarbons

1. Natural essential oils, mostly of vegetable origin.
2. Natural mineral oils -(petroleum).
3. Artificial products of destructive distillation -(paraffin oils, coaltar oils, etc.).

- (1) Lewkowitsch, J. - Chemical Technology of Oils and Fats, MacMillan and Company, London, 1913, Vol. I, Page 66.
- (2) Cowan, T.W. -Wax Craft,-Sampson, Law, Marston Company, London, 1908, Pages 15 - 44.
- (3) Wright, C.R. - Animal and Vegetable Fixed Oils, Fats, Butters and Waxes - J.B. Lippincott Co., Philadelphia, 1894, Pages 4 - 5.



4. Solid hydrocarbons obtainable from natural products (earthwax) or isolated from the two previous sources - (paraffin wax).

#### Division II - Containing Oxygen

##### A- Saponifiable

1. Essentially compound ethers of monohydric alcohols.

- (a) Various natural essential oils, mostly of vegetable origin.
- (b) Certain animal fixed oils, especially those of cetacean origin (sometimes termed "liquid waxes").
- (c) Most animal and vegetable solid waxes (waxes proper).
- (d) Certain artificial essential oils (various compound ethers used for perfumery and flavoring purposes).

2. Essentially glycerides, or compound ethers of glycerol.

- (a) The majority of animal and vegetable fixed oils, fats and butters.
- (b) Some few vegetable waxes.

##### B- Not Saponifiable

- (a) Various essential oils, consisting of aldehydes, ketones, etc.
- (b) Alcoholiform constituents of natural animal and vegetable oils (cholesterol, cetylic alcohol, etc.).
- (c) Alcoholiform bodies formed by fermentation (fusel oils).
- (d) Phenoloid bodies formed by destructive distillation and contained in coaltar (Phenol).
- (e) Products formed by oxidation of hydrocarbons - ("Sanitos oil" formed by the atmospheric oxidation of oil of turpentine).

A short outline of four other works on waxes is given here to give some idea of what has preceded this work.

Severson<sup>(4)</sup> treated the effects of various waxes on the rate of evaporation of light naphtha. In the above mentioned paper, Bees, Candelilla, Carnauba, Japan, Montan, Ozokerite and Paraffin wax were used as solutes in the solvent light naphtha. A 2% and 5% solution of each of the aforementioned waxes was prepared. One gram of the mixture was then placed on a weighed watch glass and the balance balanced. The time was recorded and a fifty gram weight was removed from the pan. The balance was allowed to remain until it again balanced and the time recorded. This was repeated until the evaporation became very slow. The amount remaining on the glass was plotted against the time taken for evaporation and compared with a curve for the pure solvent. A comparison of the evaporation of 2% and 5% solutions of the waxes was made in each case. Tables were made to show variation in rate of evaporation at different stages of drying.

A fairly complete description of the physical and chemical properties of the waxes used is given. Of the four works outlined this is the most complete description of the waxes.

Severson found that the addition of 2% and 5% of various waxes to light naphtha retarded the rate of evaporation considerably. It was also found that this rate increased depending on the amount of wax added. Candellilla wax decreased

(4) Severson, J.O. - The Effect of Various Waxes on the Rate of Evaporation of Light Naphtha, - Undergraduate Thesis.

the rate the most, while Beeswax produced the least effect. The 5% solution affected the rate of evaporation more than the 2% solution. The difference in the case of Montan was very slight.

Hable<sup>(5)</sup> studied the melting points of various binary mixtures of waxes. The introduction contains a short discussion of the chemical and physical properties of the waxes. It also contains a very brief discussion of the waxes which are classified as vegetable and animal waxes. Three procedures, the "Columbia", the "Kraemer and Sarnow", and the "Ring and Ball" methods for determining melting points are given.

In the experiments, the melting points of various binary waxes were determined by making different percentages of each pair of waxes, and determining the melting point of each mixture.

Hable found that the high melting point waxes had more influence on the melting point of a mixture than did those of low melting point. Where difference in melting points of waxes was small, the increase in the melting point of the mixture was proportional to the amount of high melting point wax present. In each case the melting point of each mixture was found to be intermediate between the melting point of the two components. The proportion of known components in the melting point mixtures yielding a straight line when plotted can be accurately calculated from the percentage composition and the melting point of each component. Carnauba wax increased the

(5) Hable, - A Study of Melting Points of Binary Mixtures of Waxes, - Undergraduate Thesis.

melting point considerably.

Spracher<sup>(6)</sup> treated the solubility of different waxes in various solvents. The introduction contained a short discussion of waxes in general. Bees, Carnauba and Paraffin wax were treated more in detail. A table on solubility of waxes was also given.

The method used in this case is as follows: The samples were weighed and placed in test tubes. The tubes were then placed in an oil bath and the wax melted, after which the hot solvent was added. The tubes were then placed in a water bath at a high temperature. They were then removed and allowed to remain in a thermostat, at constant temperature, for twenty-four hours. The solution was removed and placed in a weighed crucible and the weight of the supernatant liquid recorded. After this the crucibles were placed in an oven and the temperature kept at  $110^{\circ}$  C. for three hours. At the end of this time the solvent was evaporated and the crucibles were again weighed and the determination of the solubility calculated.

Spracher offered the curves as useful information concerning the solubility of the waxes tested. He also found that the solubility of the wax depended on the amount of solid present in equilibrium with the saturated solution.

Ford<sup>(7)</sup> studied the solubility curves of various commercial waxes in various organic solvents. In the introduction a short discussion of the physical characteristics and chemical properties of the waxes was found. A short

(6) Spracher, - Solubility of Waxes in Various Solvents, Undergraduate Thesis.

(7) Ford, L., - Solubility Curves of Various Commercial Waxes in Various Organic Solvents - Undergraduate Thesis.

classification based on saponifiable and unsaponifiable waxes was given along with a discussion of the two classes. The problem is stated as an attempt to plot curves from which it is possible to determine just how much wax is soluble in a given amount of solvent at a certain temperature. Bees, Candelilla, Carnauba, Montan, Ozakerite and Paraffin waxes were used, with chloroform and carbon tetrachloride as solvents.

The apparatus used consisted of a 125 c.c. Erlenmeyer flask fitted with a rubber stopper carrying a centigrade thermometer, a water bath and a strong source of light. One hundred grams of the solvent was placed in the flask and a weighed amount of wax added. The flask was stoppered and placed on a water bath and heated until the wax dissolved. The flask was then agitated and the solution watched closely to see at what temperature the wax began to separate. From this data the curves were plotted.

Ford found that the temperature of the room greatly affected the high concentration of the solution, causing sudden precipitation by the rapid cooling of the flask. A different result may have been obtained had the solution not been agitated. The author believes that the precipitation is continuous and gradual. The procedure used could only be used in the case of high boiling point solvents.

removed by an overflow tube which passes back down through the center of the apparatus. The whole apparatus above the stand can be turned so that the operator may have any extractor in a handy position to work with. An illustration of the extracting apparatus is shown in Figure I.

## EXPERIMENTAL APPARATUS, PROCEDURE AND RESULTS

The present paper has something in common with the works outlined in the previous chapter, and especially with the latter two. It deals with the solubility of waxes, but it differs in that it tests solubility by means of extraction.

It is known beforehand that more of the wax will be soluble by this method, because the solvent does not become saturated. When the solvent boils and condenses in the extractor it leaves the previously extracted wax in the flask. Another difference is the fact that the dissolved wax must filter through the thimble and be siphoned off. Both of these facts may have some bearing on the solubility results found in this work.

The apparatus used consisted of a 300 c.c. Erlenmeyer flask, a Soxhlet extractor, a Sargent extracting apparatus, a balance, an electric oven, and an asbestos chimney.

The Sargent extracting apparatus consists of a round hot plate large enough to hold six extractor flasks. It has a large condenser containing six tubes suitable for attaching from one to six extractors. The tubes are cooled by a stream of water which passes through a pipe in the center of the apparatus and bends over in a goose neck at the top to allow the water to drop into the condenser tank. The water is removed by an overflow tube which passes back down through the center of the apparatus. The whole apparatus above the stand can be turned so that the operator may have any extractor in a handy position to work with. An illustration of the extracting apparatus is shown in Figure I.

The asbestos chimney was made when great difficulty was experienced with freezing of the wax in the siphon tube. By means of this chimney, the heat was kept around the extractors and the wax remained in solution until siphoned. The chimney constructed was round in shape and of the same diameter as the hot plate. It was high enough to reach about half way up on the condenser tank. The asbestos was sewed together and a wire placed around it to hold it in shape. A small hole made in the asbestos chimney is very handy, as it allows the operator to observe the operation of the extractors without removing the chimney.

#### Procedure

The wax to be extracted was cut in very fine strips and weighed in the thimble which was of a known weight. The thimble was then placed in the extractor and 150 c.c. of the solvent added to a weighed flask. The flask was placed on the hot plate and the extractor attached. Each sample was allowed to remain in the extractor one and one half hours. If the extract, at the end of this time, was not clear before it siphoned over, the sample was allowed to remain until a clear extract was obtained.

When extraction was complete, the flask was removed and the larger part of the solvent evaporated off on the water bath and reclaimed. The flask was then placed in an oven at  $110^{\circ}$  C. and allowed to remain until a constant weight was obtained. From this weight the amount of wax and the percentage was determined.

In the mixtures, equal sized samples of each wax were taken. These samples were in an almost powdered form. They were then placed in an extractor and the extraction continued until complete. The flasks were then removed and the samples dried. The weight extracted was determined as above.

In the case of mixtures the amount to be extracted was calculated from the percentage obtained in the extraction of single waxes. A comparison of the calculated and actual extraction was made.

Wax	Sample	Extract	Percent
Caruba	5.162	.763	24.4
Japan	5.897	5.896	99.9
Montan	4.304	4.300	99.9
Oxokerite	5.849	1.030	17.6
Paraffin	5.4835	4.494	81.8

A gelatinous mass was left in the thistle after the extraction of Caruba and Oxokerite waxes.



TABLE NO. I  
PERCENT EXTRACTED WITH ACETONE

<u>Wax</u>	<u>Weight of Sample</u>	<u>Weight of Extract</u>	<u>Percent</u>
<u>Wax</u>	<u>Grams</u>	<u>Grams</u>	<u>Percent</u>
Bees	3.635	3.101	85.3
Candelilla	3.331	2.713	81.4
Carnauba	3.162	.763	24.4
Japan	5.897	5.896	99.9
Montan	4.384	4.300	98.08
Ozokerite	5.849	1.030	17.6
Paraffin	5.4885	4.494	81.8
Paraffin	5.223	5.166	98.7

A gelatenous mass was left in the thimble after the extraction of Carnauba and Ozokerite waxes.

TABLE NO. II

PERCENT EXTRACTED WITH BENZENE

<u>Wax</u>	<u>Weight of Sample</u>	<u>Weight of Extract</u>	<u>Percent</u>
	Grams	Grams	
Bees	4.137	4.039	97.6
Candelilla	5.197	5.174	99.5
Carnauba	4.345	4.236	97.4
Japan	3.023	3.0184	99.8
Montan	4.533	4.314	95.1
Ozokerite	2.338	2.332	99.7
Paraffin	5.229	5.166	98.7

Extreme caution must be exercised in extracting with carbon disulphide, as its flash point is very low and the heat of the hot plate might ignite it. The solvent in the above case was allowed to evaporate without heat being applied.

TABLE NO. III

PERCENT EXTRACTED WITH CARBON DISULPHIDE

<u>Wax</u>	<u>Weight of Sample</u> Grams	<u>Weight of Extract</u> Grams	<u>Percent</u>
Bees	3.637	3.624	99.6
Candelilla	3.426	3.409	99.2
Carnauba	3.951	3.8566	97.6
Japan	4.5106	4.5100	99.9
Montan	4.160	4.141	99.5
Ozokerite	3.2125	3.2115	99.9
Paraffin	8.024	8.022	99.9

Extreme caution must be exercised in extracting with carbon disulphide, as its flash point is very low and the heat of the hot plate might ignite it. The solvent in the above case was allowed to evaporate without heat being applied.

TABLE NO. IV

PERCENT EXTRACTED WITH CARBON TETRACHLORIDE

<u>Wax</u>	<u>Weight of Sample</u>	<u>Weight of Extract</u>	<u>Percent</u>
	Grams	Grams	Percent
Bees	3.254	3.2205	98.9
Candelilla	3.9307	3.8152	97.9
Carnauba	5.718	5.6746	99.2
Japan	5.539	5.410	97.5
Montan	4.0675	4.0635	99.7
Ozokerite	12.459	12.456	99.9
Paraffin	13.940	13.813	99.09

TABLE NO. V  
PERCENT EXTRACTED WITH CHLOROFORM

<u>Wax</u>	<u>Weight of Sample</u>	<u>Weight of Extract</u>	<u>Percent</u>
	Grams	Grams	
Bees	3.948	3.947	99.9
Candelilla	6.3312	6.2906	96.2
Carnauba	2.199	2.123	96.5
Japan	4.976	4.974	99.9
Montan	3.2434	3.161	97.7
Ozokerite	3.316	3.309	99.7
Paraffin	5.2574	5.2389	99.6

With Candelilla, Carnauba and Ozokerite wax a white  
soggy mass was left in the residue. The wax seemed to be  
coated with a white soft coat still the residue was more or  
less solid.

TABLE NO. VI  
PERCENT EXTRACTED WITH ETHER

<u>Wax</u>	<u>Weight of Sample</u> Grams	<u>Weight of Extract</u> Grams	<u>Percent</u>
Bees	3.800	3.139	82.6
Candelilla	3.766	2.411	64.02
Carnauba	2.776	.262	9.4
Japan	4.793	4.564	95.2
Montan	4.268	3.5184	82.4
Ozokerite	3.852	.950	24.6
Paraffin	6.066	6.053	99.7
Paraffin	3.487	3.487	97.4

With Candelilla, Carnauba and Ozokerite wax a white soggy mass was left in the thimble. The wax seemed to be coated with a white soft coat while the center was more or less solid.

TABLE NO. VII

EXTRACTION PERCENT EXTRACTED WITH NAPHTHA

Wax	Mixture	Weight of	1st	Weight of	Calculated	Actual
		Sample		Extract		
1st	2nd	Grams	Percent	Grams	Grams	Grams
Bees	-Candelilla	4.736	98.9	4.623	97.6	5.912
Candelilla	-Carnauba	4.048	98.9	3.739	92.1	5.762
Carnauba	-Japan	5.111	98.9	2.110	41.08	5.997
Japan	-Montan	4.494	98.9	4.315	96.2	5.998
Montan	-Ozokerite	4.151	98.9	3.469	83.5	5.968
Ozokerite	-Paraffin	3.827	98.9	3.674	96.00	5.991
Paraffin	-Carnauba	3.487	97.9	3.407	97.4	3.809
Candelilla	-Japan		97.9	97.5	5.862	5.830
Candelilla	-Montan		97.9	99.7	5.939	5.963
Carnauba	-Ozokerite		99.2	99.9	5.952	3.901
Carnauba	-Paraffin		99.2	99.09	3.956	3.740
Japan	-Montan		99.4	99.7	5.973	5.996
Japan	-Ozokerite		99.4	99.9	5.979	5.913
Japan	-Paraffin		99.4	99.09	3.986	3.800
Montan	-Ozokerite		99.7	99.9	5.988	5.97
Montan	-Paraffin		99.7	99.09	5.9637	5.953
Ozokerite	-Paraffin		99.9	99.09	5.9732	3.938

A large part of the sample remained in the thimble in the case of Carnauba wax. The extraction with Naphtha went very slow and added heat was applied to force the naphtha to pass into the extractor.

TABLE NO. VIII

EXTRACTION OF 50-50-MIXED WAXES WITH CARBON TETRACHLORIDE

<u>50-50 Wax Mixtures</u>		<u>Extraction of 1st</u>	<u>Extraction of 2nd</u>	<u>Calculated Extract</u>	<u>Actual Extract</u>
1st	2nd	Percent	Percent	Grams	Grams
Bees	-Candelilla	98.9	97.9	5.904	5.912
Bees	-Carnauba	98.9	99.2	5.943	5.752
Bees	-Japan	98.9	97.5	5.892	5.997
Bees	-Montan	98.9	99.7	5.958	5.998
Bees	-Ozokerite	98.9	99.9	5.964	5.968
Bees	-Paraffin	98.9	99.09	5.9397	5.921
Candelilla-Carnauba		97.9	99.2	3.932	3.899
Candelilla-Japan		97.9	97.5	5.862	5.850
Candelilla-Montan		97.9	99.7	5.928	5.983
Candelilla-Ozokerite		97.9	99.9	3.956	3.965
Candelilla-Paraffin		97.9	99.09	5.9097	5.983
Carnauba	-Japan	99.2	97.5	5.901	5.856
Carnauba	-Montan	99.2	99.7	5.987	5.877
Carnauba	-Ozokerite	99.2	99.9	3.982	3.901
Carnauba	-Paraffin	99.2	99.09	3.9658	3.740
Japan	-Montan	99.4	99.7	5.973	5.996
Japan	-Ozokerite	99.4	99.9	5.979	5.913
Japan	-Paraffin	99.4	99.09	3.986	3.800
Montan	-Ozokerite	99.7	99.9	5.988	5.97
Montan	-Paraffin	99.7	99.09	5.9637	5.963
Ozokerite	-Paraffin	99.9	99.09	3.9798	3.968



TABLE NO. IX

EXTRACTION OF 50-50-MIXED WAXES WITH ACETONE

<u>50-50 Wax Mixtures</u>		<u>Extraction of 1st</u>	<u>Extraction of 2nd</u>	<u>Calculated Extract</u>	<u>Actual Extract</u>
1st	-2nd	Percent	Percent	Grams	Grams
Bees	- Carnauba	85.3	24.4	2.194	2.501
Bees	- Paraffin	85.3	81.8	3.342	3.989
Carnauba	- Paraffin	24.4	81.8	2.124	2.488

This and the following tables are incomplete. Time prohibited the extraction of all mixtures; therefore the three most common waxes are used in this and the following tables.

TABLE NO. X

EXTRACTION OF 50-50-MIXED WAXES WITH BENZENE

<u>50-50 Wax Mixtures</u>		<u>Extraction of 1st</u>	<u>Extraction of 2nd</u>	<u>Calculated Extract</u>	<u>Actual Extract</u>
1st	2nd	Percent	Percent	Grams	Grams
1st	2nd	Percent	Percent	Grams	Grams
Bees	- Carnauba	97.6	97.4	3.900	3.933
Bees	- Paraffin	97.6	98.7	3.926	3.985
Carnauba-	Paraffin	97.4	98.7	3.922	3.9052
Carnauba	- Paraffin	97.6	98.9	3.950	3.945

TABLE NO. XI

EXTRACTION OF 50-50-MIXED WAXES WITH CARBON DISULPHIDE

<u>50-50 Wax Mixtures</u>		<u>Extraction of 1st</u>	<u>Extraction of 2nd</u>	<u>Calculated Extract</u>	<u>Actual Extract</u>
1st	2nd	Percent	Percent	Grams	Grams
Bees	- Carnauba	99.6	97.6	3.944	4.060
Bees	- Paraffin	99.6	99.9	3.990	4.032
Carnauba	- Paraffin	97.6	99.9	3.950	3.945

TABLE NO. XII

EXTRACTION OF 50-50-MIXED WAXES WITH CHLOROFORM

<u>50-50 Wax Mixtures</u>		<u>Extraction of 1st</u>	<u>Extraction of 2nd</u>	<u>Calculated Extract</u>	<u>Actual Extract</u>
1st	2nd	Percent	Percent	Grams	Grams
Bees	- Carnauba	99.9	96.5	3.982	3.939
Bees	- Paraffin	99.9	99.6	3.990	3.889
Carnauba	- Paraffin	96.5	99.6	3.922	3.934

TABLE NO. XIII

EXTRACTION OF 50-50-MIXED WAXES WITH ETHER

<u>50-50 Wax Mixtures</u>		<u>Extraction of 1st</u>	<u>Extraction of 2nd</u>	<u>Calculated Extract</u>	<u>Actual Extract</u>
1st	2nd	Percent	Percent	Grams	Grams
Bees	- Carnauba	82.6	9.4	1.840	2.228
Bees	- Paraffin	82.6	99.7	3.834	3.985
Carnauba	- Paraffin	9.4	99.7	2.182	2.293

THE EXTRACTION OF POLISH

TABLE NO. XIV

EXTRACTION OF 50-50-MIXED WAXES WITH NAPHTHA

The polish used is a commercial product. It is a water emulsion, is non-inflammable and is applied by rubbing or polishing. The polish was applied by the ordinary wax finish upon drying. It is claimed that no rubbing or polishing is necessary. The polish was applied by

<u>50-50 Wax Mixtures</u>		<u>Extraction of 1st</u>	<u>Extraction of 2nd</u>	<u>Calculated Extract</u>	<u>Actual Extract</u>
1st	2nd	Percent	Percent	Grams	Grams
Bees	- Carnauba	97.6	41.08	2.8168	3.989
Bees	- Paraffin	97.6	97.4	3.900	3.990
Carnauba	- Paraffin	41.08	97.4	2.7696	3.942

and the one extracted with absolute alcohol and carbon tetrachloride, in the order named. The percent extracted with each solvent was then determined, and also the percent of unextracted material. The acid, saponification and iodine number was also determined in the two extracts. The other sample was extracted with carbon tetrachloride and absolute alcohol in the order named, and the same procedure as above carried out.

A test for shellac in the polish was also made. A sample of the polish was taken and evaporated to dryness in the oven. It was then placed on a hot plate and melted. When all the solid had melted it was stirred with a glass rod. The shellac adhered to the rod and also to the sides of the glass container.

THE EXTRACTION OF FURNITURE POLISH

The polish used is a commercial product. It is a water emulsion, is non-inflamable and is subject to freezing. The producers claim that the polish can be applied and will give the ordinary wax finish upon drying. It is claimed that no rubbing or polishing is necessary. The polish was applied by the author and the results were not as good as expected.

In this work a sample of the polish was taken and evaporated to dryness. The sample was then divided into two parts and the one extracted with absolute alcohol and carbon tetrachloride, in the order named. The percent extracted with each solvent was then determined, and also the percent of unextracted material. The acid, saponification and iodine number was also determined in the two extracts. The other sample was extracted with carbon tetrachloride and absolute alcohol in the order named, and the same procedure as above carried out.

A test for shellac in the polish was also made. A sample of the polish was taken and evaporated to dryness in the oven. It was then placed on a hot plate and melted. When all the solid had melted it was stirred with a glass rod. The shellac adhered to the rod and also to the sides of the glass container.

Saponification Number of Extract.....	348.295
Iodine Number of Extract.....	2.3885
Weight of Unextracted Material.....	2.421
Percent of Unextracted Material.....	15.1

FURNITURE POLISH DATA

Weight of Sample No. I .....	14.954	Grms.
Weight Extracted with Absolute Alcohol.....	5.592	Grms.
Percent Extracted with Absolute Alcohol.....	37.4	%
Acid Value of Extract.....	13.34	
Saponification Number of Extract.....	322.425	
Iodine Number of Extract.....	4.6726	
Weight Extracted with Carbon Tetrachloride.....	6.1205	Grms.
Percent Extracted with Carbon Tetrachloride.....	40.9	%
Acid Value of Extract.....	4.718	
Saponification Number of Extract.....	222.13	
Iodine Number of Extract.....	1.1826	
Weight of Unextracted Material.....	3.2415	Grms.
Percent of Unextracted Material.....	21.6	%
Weight of Sample No. II .....	14.947	Grms.
Weight Extracted with Carbon Tetrachloride.....	11.365	Grms.
Percent Extracted with Carbon Tetrachloride.....	76.04	%
Acid Value of Extract.....	26.45	
Saponification Number of Extract.....	251.305	
Iodine Number of Extract.....	2.333	
Weight Extracted with Absolute Alcohol.....	1.161	Grms.
Percent Extracted with Absolute Alcohol.....	7.7	%
Acid Value of Extract.....	6.01	
Saponification Number of Extract.....	342.295	
Iodine Number of Extract.....	2.3888	
Weight of Unextracted Material.....	2.421	
Percent of Unextracted Material.....	16.1	

1918, Volume II, Page 123.

(9) Ryan, P. - Oils, Fats and Waxes, -Vol. II, Pages 124-26

(10) Ryan, P. - Oils, Fats and Waxes, -Vol. II, Page 106



## THE EXTRACTION OF SHELLAC

Flake shellac was taken and divided into two samples. They were placed in an extractor and the first extracted with absolute alcohol and carbon tetrachloride in the order named. The percent extracted with each solvent was then determined and also the percent of unextractable material. The acid, saponification and iodine numbers of each were then determined. The other sample was extracted with 95% alcohol and carbon tetrachloride, and the same procedure as above carried out.

### Acid Value

"The acid value of an oil, fat, or wax is a measure of the free fatty acidity, and represents the number of milligrammes of potassium hydroxide required to neutralise the free fatty acids in 1 gramme of the substance." (8)

The procedure used in the determination of the acid value of both shellac and polish is the same. The procedure is that outlined in Fryer<sup>(9)</sup> and is in short as follows. The sample is taken and dissolved in about 50 c.c. of boiling neutral alcohol. Phenolphthalein is added as an indicator and the solution titrated with a  $\frac{N}{2}$  alkali solution.

$$\text{Acid Value} = \frac{\text{No. of c.c. used} \times 28.05}{\text{Weight of sample taken}} \quad (\text{with } N/2 \text{ alkali})$$

### Saponification Value

"The saponification value indicates the number of milligrammes of potassium hydroxide required to completely saponify one gramme of the substance." (10)

(8) Fryer, P.- Oils, Fats and Waxes, -G.P. Putnam's Sons, N.Y. 1918, Volume II, Page 123.

(9) Fryer, P.- Oils, Fats and Waxes, -Vol.II, Pages 124-25

(10) Fryer, P.- Oils, Fats and Waxes, -Vol.II, Page 106

The procedure used in the determination of the saponification value of shellac and polish is the same. This procedure is also taken from Fryer.<sup>(11)</sup> A sample is taken and 25 c.c. of alcoholic potash added. It is then placed under a reflux and boiled for thirty minutes. 10 c.c. of neutralized alcohol is then added, with phenolphthalein as an indicator. The solution is then titrated with N/2 hydrochloric acid solution. A blank determination was also run and the saponification value calculated.

Saponification Value =

$$\frac{(\text{c.c. used for blank} - \text{c.c. used for wax}) \times 28.05}{\text{Weight of sample taken}}$$

#### Iodine Value

"The iodine value is the percentage of iodine chloride expressed in terms of iodine which is absorbed by an oil, fat or wax." (12)

The procedure used in iodine value determination as outlined in Fryer<sup>(13)</sup>. But for the shellac, it was found impossible to dissolve the extract in carbon tetrachloride. At the suggestion of Mr. Fry of Patek Brothers Paint Company, glacial acetic acid was tried and found to work very well. The sample was taken and dissolved in carbon tetrachloride (shellac in glacial acetic acid). 10 c.c. of Wijs' solution was added and the solution allowed to stand thirty minutes.

(11) Fryer, P.- Oils, Fats and Waxes, -G.P. Putnam's Sons, N.Y. 1918, Volume II, Page 108.

(12) Fryer, P.- Oils, Fats and Waxes, -Vol.II, Page 92

(13) Fryer, P.- Oils, Fats and Waxes, -Vol.II, Pages 94 - 95.

10 c.c. of potassium iodide solution and 50 c.c. of water were then added and the solution titrated with N/10 sodium thiosulphate solution. A blank determination was also run.

Iodine Value =

Weight of Sample No. I.....	8.87	Grms.
(c.c. used for blank - c.c. used for wax) x .012692 x 100		x factor
<hr/>		
Percent Extracted with Weight of sample taken.....	73.8	%

Acid Value of Extract.....	206.7	
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All reagents and solutions used in the acid, saponification and iodine value determinations were prepared according to the method outlined in Fryer.

Saponification Number.....	282.7	
Iodine Number of Extract.....	1.789	
Weight Extracted with Carbon Tetrachloride.....	.484	Grms.

Percent Extracted with Carbon Tetrachloride.....	5.8	%
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Weight of Unextracted Material.....	1.7823	Grms.
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Percent of Unextracted Material.....	20.0	%
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Weight of Sample No. II.....	10.0458	Grms.
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Weight Extracted with 95% Alcohol.....	5.1185	
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Percent Extracted with 95% Alcohol.....	50.9	%
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Acid Value of Extract.....	272.5	
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Saponification Value of Extract.....	386.5	
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Iodine Value of Extract.....	3.062	
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Weight Extracted with Carbon Tetrachloride.....	.2748	Grms.
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Percent Extracted with Carbon Tetrachloride.....	2.5	%
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Weight of Unextracted Material.....	5.9188	Grms.
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Percent of Unextracted Material.....	59.3	%
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SHELLAC DATA

1. The author found that Acetone and Ether were the two

Weight of Sample No. I.....	8.67	Grms.
Weight Extracted with Absolute Alcohol.....	6.4035	Grms.
Percent Extracted with Absolute Alcohol.....	73.8	%
Acid Value of Extract.....	206.7	
Saponification Number.....	282.7	
Iodine Number of Extract.....	1.769	
Weight Extracted with Carbon Tetrachloride.....	.484	Grms.
Percent Extracted with Carbon Tetrachloride.....	5.5	%
Weight of Unextracted Material.....	1.7825	Grms.
Percent of Unextracted Material.....	20.5	%
Weight of Sample No. II.....	10.0455	Grms.
Weight Extracted with 95% Alcohol.....	5.1185	
Percent Extracted with 95% Alcohol.....	50.9	%
Acid Value of Extract.....	272.3	
Saponification Value of Extract.....	355.5	
Iodine Value of Extract.....	2.066	
Weight Extracted with Carbon Tetrachloride.....	.9742	Grms.
Percent Extracted with Carbon Tetrachloride.....	9.6	%
Weight of Unextracted Material.....	3.9488	Grms.
Percent of Unextracted Material.....	39.3	%

2. Carbon Disulphide and Carbon Tetrachloride showed the  
most solvent powers.

3. Japan and Paraffin waxes were very soluble in all of the  
solvents.

4. It was found that Shellac was not completely extractable  
with 95% or absolute alcohol. However, it was more ex-  
tractable with absolute alcohol than with 95% alcohol.

5. A glass rod was found to be a fairly good test for the  
presence of Shellac in a mixture of waxes.

SUMMARY

3. An aspirator chimney was used to great advantage in
1. The author found that Acetone and Ether were the two poorest solvents for the single waxes.
2. Carbon Disulphide and Carbon Tetrachloride showed the most solvent powers.
3. Carnauba wax was for the most part the least soluble of the waxes tried.
4. Japan and Paraffin waxes were very soluble in all of the solvents.
5. In the mixed waxes it was found that the actual and calculated weights which were extracted compared fairly well. In most cases, however, the actual weight exceeded the calculated weight to a very slight degree. The greatest increases of the actual over the calculated weight were found when Bees and Carnauba were extracted with Aceton, when Bees and Carnauba were extracted with Ether, and when Carnauba and Paraffin were extracted with Naphtha.
6. It was found that Shellac was not completely extractable with 95% or absolute alcohol. However, it was more extractable with absolute alcohol than with 95% alcohol.
7. A glass rod was found to be a fairly good test for the presence of Shellac in a mixture of waxes.

8. An asbestos chimney was used to great advantage in speeding up extraction and in guarding against frozen siphon tubes in the Soxhlet extractors.

"Wool Grease" - Sampson, Low, London, 1913.  
The book contains the history of wool in general, the origin, production, rearing, raising and cleaning of fleeces. It also treats the adulteration and technical uses of wool.

WATER, P. I.

"Technical Handbook of Oils, Fats and Greases" - I.F. Fennell's Edn. The Chem. Soc., 1914.

This book is in two volumes. It covers the production, analysis, classification, purification, refinement and adulteration of the oils, fats and greases. It also deals with the properties of these materials. It gives methods and procedures for determining the specific gravity, melting point, boiling point, refractive index, viscosity, saponification value, iodine value, acid value, saponification value, insoluble residue value, oxidation value, etc. It also contains a list of symbols and abbreviations.

WATER, P. II.

"The Industrial Chemistry of the Fats and Greases" - Ed. by Tinsall and Cox, London, 1927.

This book contains the chemical nature of fats and their occurrence in nature, and the industrial uses of fats and greases in paints and varnishes.

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"Wax Craft" - Sampson, Low, Marston Company, London, 1908.  
The book contains the history of wax in general, the origin, production, rendering, refining and bleaching of Beeswax. It also treats the adulteration and technical uses of wax.

FRYER, P.J.

"Technical Handbook of Oils, Fats and Waxes" - G.P. Putnam's Sons, New York, 1918.

The book is in two volumes. It treats the chemistry, analysis, classification, production, refinement and adulteration of the oils, fats and waxes. It also deals with the preparation of samples for analysis. It gives methods and procedures for determining the Specific gravity, Melting point, Solidifying point, Refractive power, Viscosity, Solubility, Rotatory power, Iodine value, Elaidin test, Saponification value, Insoluble Bromide value, Reichert-Meissel value, Acetyl value, Acid value and Un-saponifiable matter. It also contains a list of tables and illustrations.

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"The Oil-Chemists' Handbook" - First Edition - John Wiley and Sons, New York, 1900.

This book contains the general properties of the waxes and a very complete list of tables on oils, fats and waxes.

LEWKOWITSCH, J.

"Chemical Technology and Analysis of Oils, Fats and Waxes" - MacMillan and Company, London, 1923.

This book is in three volumes, and contains the classification, chemical and physical properties of oils, fats and waxes. It also contains the constituents of the oils, fats and waxes and methods and procedures for analysis. The book gives a complete list of tables on the oils, fats and waxes.

WRIGHT, C.R.

"Animal and Vegetable Fixed Oils, Fats, Butters and Waxes" - J.B. Lippincott Company, Philadelphia, 1894.

This book contains the general composition, sources, physical and chemical properties of the oils, fats, butters and waxes. It also gives the reactions of the oils, fats, butters and waxes. Processes for refining, rendering, extracting and bleaching are treated. It gives a complete classification, with uses and adulterations of the oils, fats, butters and waxes. The candle and soap industries are treated fully.

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APPROVED

John R Koch

Major Professor

W. J. Grace, Jr.

Dean

Date

May 4, 1932.