

SWANN RESEARCH INC.
DEVELOPMENT DEPARTMENT
PROJECT NO 141
CHLORINATED DIPHENYLS (AROCLORS)
Final Reports
October 1950
Mr. J. L. Howerton
Mr. J. W. Perry Jr.
Mr. J. E. Malowan

0288578

S. SWANN RESEARCH INC.,

DEVELOPMENT DEPARTMENT

PROJECT NO 141

.CHLORINATED DIPHENYLS (AROCLORS)

COMPLETE REPORT

November 1, 1950

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SWANN RESEARCH INC.

DEVELOPMENT DEPARTMENT

PROJECT NO 141

COMPLETE REPORT ON AROCLORS

This report includes a complete description of the work done in this department on Aroclors. It is made up of 3 separate reports, the first by Mr. J. L. Bowerton describing distillation, both as to its development and operation. He also describes the operation of the plant chlorinators.

The other reports deal with work on chlorination. It was soon made obvious that in order to get a uniform distilled product, it was necessary to chlorinate under much more carefully controlled conditions than was then in vogue in the plant.

The second report by Mr. J. G. Perry Jr., clearly shows that low temperature and accurate temperature control is absolutely essential to product uniformity. Quite as important is uniform chlorine addition and positive agitation. After reading his report you can readily see that a powdered iron catalyst has obvious advantage over a receptacle filled with particles of iron. Please understand that while much is said about the yield of 4-4' dichlor-diphenyl, this was largely incidental. This compound could be more readily isolated than others, and was therefore used as the indicator or yard stick on the experimentation. It so happened that we were also working toward a maximum yield of 4-4' dichlor-diphenyl at this time.

Report #3 represents the line of work done by Mr. J. E. Malowan. His objective was largely a maximum yield in chlorination of 4-4' dichlor-diphenyl. Incidentally a lot of most valuable information on the whole phenomena was brought out. His work again shows very conclusively the basic importance of low temperature chlorination as well as temperature control, and throws a strong light on the reason for poor yields of various isomers under various conditions.

W. R. Seyfried

WRS

WRS/jw

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DEVELOPMENT OF THE MANUFACTURE OF VACUUM

DISTILLED ARCCLORS

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 Distilled Aroclors

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Blueprint	2	Distillation Range of Aroclor	1242
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"	4	"	"
"	5	"	"
"	6	"	"
"	7	"	"
"	8	"	"
"	9	"	"
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SWANN RESEARCH INC

DEVELOPMENT DEPARTMENT

PROJECT 141

DEVELOPMENT OF MANUFACTURE OF VACUUM

DISTILLED AROCLORS

Final Report Oct. 16, 1930

Summary

After approximately twelve months work beginning in the summer of 1929 and ending the following summer the Development Department has developed a process and designed equipment for the large-scale manufacture of vacuum-distilled Aroclors.

In this report the work done and difficulties encountered are discussed, and the steps shown by which the process was finally developed.

Estimates of manufacturing costs both on the present and on large-scale equipment have previously been prepared and submitted, and are therefore not included in this report. However, it has been found that these costs are low enough to enable us to manufacture the Aroclors and sell them at reasonable prices.

To date, judging by the interest shown by prospective users, the Aroclors rank in order of importance as follows: 1254, 1262, 4455, 1242, 1248, 1264, and 1268. The first five are far ahead of the last two and there is little to choose between the last two.

History

After considerable work both on chlorination and distillation the Research Department submitted this problem to the Development Department in the summer of 1929. The Research Department had prepared and submitted samples to prospective customers and sufficient interest had been displayed to warrant the development of a manufacturing process. However, until we attained a semi-manufacturing basis in the last stages of this work, the development work was always handicapped by the necessity of preparing samples while developing the process. While this was irksome at the time, it is probable that had not these samples been submitted as the work progressed the interest displayed in these products would not as yet have been pronounced.

This work falls into three stages as follows:

- (1) First crude still which was unsuccessful, but which served as a guide in avoiding future pit-falls.
- (2) Design, construction, and operation of the Buford Still. This equipment was only partially successful and was finally abandoned, but it represents a valuable stage in the evolution of the process.

- (3) Purchase of the tube still from E. B. Badger & Sons. This unit was not successful as purchased, but after many changes was found to be the proper type of still for the manufacture of these products.

DESCRIPTION OF THE ARCELORS

Arcelors manufactured

The Arcelors covered in this report include 1242, 1248, 1254, 1262, 1238, and 4405. Arcelor 1219 is not included since it is not a vacuum-distilled product.

Nomenclature

With the exception of Arcelor 4465 the digits in the numbers have the following significance: The first number indicates that before chlorination the product consisted of 100% technical diphenyl; the second number shows the number of operations that have been performed; if "1", e.g. 1142, the product has been chlorinated, if "2", e.g. 1242, the product has been both chlorinated and distilled. The last two figures show that the product after chlorination contains that percentage of chlorine. In the case of Arcelor 4465, the first digit indicates that the product contained 40% distilled high boiler, and subtracting this percentage from 100%, 60% technical diphenyl. The second figure, if "0" e. g. 4035, shows chlorination while if "4", e. g. 4465, shows both chlorination and distillation. The last two figures show Chlorine content.

All of this series of products are polychlorinated diphenyls. They are not Homogeneous compounds, but are probably mixtures not only of several isomers but of several compounds containing varying percentages of chlorine. Therefore an Arcelor will not have a boiling point but a boiling range. The specific gravity of the Arcelors varies in proportion to the chlorine content.

Arcelor 1212

This is a comparatively light oil which corresponds in chlorine percentage to the formula $C_{12}H_7Cl_3$ or tri-chloro-diphenyl, although if fractionated it would yield compounds of higher and lower chlorine percentage.

The specifications for this Arcelor are as follows:

Specific Gravity	1.360 $\pm$ 0.005 @ 55°/65° C
Free Acid	Not more than 0.02 mg./NaOH/ gm. material
Color	Water-white to pale yellow

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Arcolor 1242 (Specifications Cont.)

Sediment	None
Chlorine Content	41.5% - 42.5%
Viscosity	35.2 ± 0.9 seconds Saybolt at 210° F.
Distillation Range	324° - 360° C
Flash Point	174°C
Flame Point	224°C
Iron	Not more than 0.02 p. p. m.

Arcolor 1248 *

The viscosity of this Arcolor approximates that of a heavy motor oil, and approximates the formula $C_{12}H_9Cl_4$ or tetra-chloro diphenyl. This material has been prepared especially for the Constock & Westcott Co., Niagara Falls, N. Y., to comply with a specified boiling range. Its physical characteristics other than boiling range have not been determined. The specifications are as follows:

Specific Gravity	1.42 ± 0.005 at 65°/65° C
Chlorine Content	47.5% - 49.5%
Free Acid	Not more than 0.02 mg. NaOH/ gm. material
Iron Content	Not more than 2 p. p. m.
Color	Water white to pale lemon yellow
Sediment	None
Boiling Range	90° - 345° - 360°C 104° - 360° - 370°C

Arcolor 1254

This is a viscous oil which approximates the formula $C_{12}H_5Cl_5$ or penta- chloro diphenyl. Its specifications are as follows:

Specific Gravity	1.520 ± 0.005 at 65°/65° C
Free Acid	Not more than 0.02 mg./NaOH/gm. material
Sediment	None
Color	Pale lemon yellow
Chlorine Content	54.5% - 55.5%
Iron Content	Not more than 2 p. p. m.
Viscosity	46.7 ± 0.9 seconds Saybolt at 210°F
Flash Point	210°C (Indistinct)
Flame Point	None up to boiling point
Distillation Range	360° - 405° C.

* The name "1248" given this Arcolor is only tentative and is subject to change. It has been suggested that it be called Arcolor 1248 to denote a special product.

When the still is being operated continuously on the same Aroclor still residues may be allowed to accumulate until the sixth day. On that day and every day thereafter 100 lbs. should be withdrawn at the end of a run. This will keep the contents of the still at approximately two-thirds of its capacity since it will hold 2100 lbs. and the residue will accumulate at the rate of approximately 100 lbs. per day.

When a change is made from one Aroclor to another all residues should be withdrawn. Extreme precaution must be taken during this operation to prevent freezeups. The bottoms are pumped out through the globe valve located just below the coils, at the same time closing the valve leading to the coils. Simultaneously the vacuum is exhausted through the coils by breaking the union at the coil inlet. Immediately after this operation before the pump has time to freeze the new hot material is charged and circulation started. When the still is to be shut down for an indefinite period this operation is repeated but at the same time a plug at the foot of the pump is removed and the pump drained thoroughly by running it for ten or fifteen minutes or until empty.

In the distillation of Aroclor 4465 the operator must be careful not to stop the distillation too quickly. Since this Aroclor contains two components the lower boiling portion will come over first; there will then be a lag in the distillation rate and vapor temperature. The stream and vapor temperature will again increase as the higher boiling portion vaporizes and the end of the run will be unmistakable. A study of the distillation curve for this Aroclor given in the appendix will reveal the peculiarities of this distillation.

DEVELOPMENT OF PROCESS

Theory of Distillation

Since all the Aroclors are mixtures of several components containing varying percentages of chlorine and having different boiling points, the object of all this work was to develop apparatus which could distill the material with a minimum of fractionation but with maximum prevention of frothing or entrainment of carbonaceous or tarry matter contained in the crude chlorinated Aroclor. Thus a short but highly efficient fractionating column which would carry the vapor rapidly away from the heating zone and would give little or no reflux was necessary. Another advantage of quick removal of vapor from the heating zone lies in the consequent lowering of decomposition.

The Research Department had found that all of the Aroclors with the exception of 4465 could be distilled at atmospheric pressures but that, as is the case with any organic compound, the longer heating period and higher temperature required for such distillation would result in excessive decomposition. Therefore, distillation at reduced pressures was also necessary for these.

<u>Arcolor No.</u>	<u>Lbs. Chgd.</u>	<u>Lbs. Dist.</u>	<u>Lbs. S. B.</u>	<u>% Recovery</u>
1242	8337	8114	220	97.09
1248	9300	3700		40.00 +
1254	58700	58782	1918	95.04
1262	12990	12201	659	95.93
1264	1935	1620	300	83.72
1268	1050	900	150	85.71
4465	27404	23952	3400	87.43 +
Totals	99736	87269	6647	

All of these figures are based on the amount of material charged to the still and not on that charged to the chlorinator. When chlorination losses are taken into account the recoveries will average 5% less than these.

Improvement of Product

A comparison of analytical results between the earliest and latest batches distilled with lime show a marked improvement in the quality of the product, since it has been found lately that iron and free acid have a marked effect on the electrical properties of the Arcolors as well as the color, our efforts during the latter part of the development work have been bent toward eliminating these impurities. The use of hydrated lime in the still charge and the replacement of iron equipment by monel have enabled us to practically accomplish this end. Iron was not determined on the earliest batches but it has been found that this element which is present as ferric chloride, will titrate as free acid. Therefore the free acid content is indicative of the iron present. Following are comparisons of these analyses:

Arcolor 1242

	<u>Early Batch</u>	<u>Late Batch</u>
p. Cr. 65°/65°C	1.933	1.761
Chlorine		
Acidity	0.0192	0.0009
Iron (p.p.m.)	1.8	0.5

Arcolor 1254

p. Cr. 65°/65°C	1.521	1.520
Acidity	0.0123	0.00085
Iron (p. p. m.)	Not det.	0.4

* This figure includes only that material produced within the boiling range. In estimating costs the balance of the distillate is credited to the operation at raw material prices.

* * This is an average figure. The latest batches distilled show 90% recovery, and this figure can be maintained on regular operation.

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Distilled Arcelors
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Arcelor 1242	2500 lbs.
" 1243	600 "
" 1254	2500 "
" 1262	2500 "
" 1264	2200 "
" 4465	2000 "

This assumes continuous operation and should be shaded by approximately 500 lbs. each to allow for shut-downs and repairs.

Large Scale Equipment

From the experience gained and data obtained during this work, Mr. K. A. Kilbourne has designed an operating unit of approximately five times the capacity of the present unit. This equipment has been drawn up in detail by the Engineering Department and a preliminary estimate made of its cost so that when sales prospects justify its construction we will be ready for the manufacture of the Arcelors within a comparatively short time. In the meantime the present unit should suffice to produce our requirements during the next six months.

Conclusion:

It is believed by the writer that this report demonstrates the fact that we have developed a satisfactory process for the production of high quality Arcelors. Besides the writer the following men were identified with the development of the process:

Mr. K. A. Kilbourne
Mr. M. N. Gaboury
Mr. J. W. Perry Jr.
Mr. J. E. McLoven
Dr. Rogers McCullough
Mr. E. H. Buford
Mr. W. N. Seyfried

Without the assistance of these men the project would not have been nearly as far advanced as it is as all have contributed invaluable advice and assistance.

J. L. Howerton
J. L. Howerton

JLR/jw

WRS

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TABLE 1 - HOURLY SAMPLES - BATCH 21-A - AROCLOR 1254

<u>Time</u>	<u>T° Coil</u>		<u>T° Vapor</u>	<u>Pressure-lb.</u>		<u>Sp.Gr. 65/65 °C</u>	<u>% Cl</u>	<u>Acidity</u>	<u>Fe (P.P.M)</u>
	<u>Inlet</u>	<u>Outlet</u>		<u>Kettle</u>	<u>Receiver</u>				
5:30 AM	160°C	202°C	203°C	5	5	1.498	53.31	0.018	-
6:30	172	217	203	5	5	1.496	53.21	0.009	-
8:00	178	223	210	8	6	1.501	53.57	0.005	-
9:00	184	235	213	9	6	1.507	54.03	0.004	-
10:00	188	242	218	8	6	1.515	54.68	0.005	-
11:00	191	246	221	8	6	1.525	55.25	0.005	-
12:00 M	195	250	224	9	6	1.535	56.15	0.004	-
1:00 PM	198	256	226	9	6	1.550	57.01	0.005	-
2:00	209	261	230	9	7	1.565	58.43	0.006	-
3:00	230	280	235	11	8	No Sample - Run Complete			

Analysis 1154 Charged

Sp.Gr. @ 65/65°C 1.527
 % Cl 55.52
 Acidity 0.02 Mg. NaOH/Gm. Material

Analysis Whole Batch

Sp.Gr. @ 65/65° C 1.525
 Acidity 0.005
 % Chlorine Not Determined
 Fe " "

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RESEARCH LABORATORY

Swann Research, Incorporated.

Anniston, Alabama.

PRELIMINARY SURVEY REPORT

ARCOLOGS IN PHONOGRAPH RECORDS

(Victor Talking Machine Company
of Canada.)

By

R. L. Jenkins.
(May 18, 1930.)

Six copies of this report were made, and distributed as follows:-

No.1- File
No.2- J.H. Carothers
No.3- R.C. Munn
No.4- W.H. Seyfried
No.5- R.H. Coward
No.6- Paul Logue

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OBJECT.

To determine whether Research work on Arcolors for the Victor Talking Machine Company of Canada is warranted; and if so, what line of attack should be pursued.

SUMMARY & CONCLUSIONS:

All of the information available in the Anniston files on the subject of the use of Arcolors by the Victor Talking Machine Company of Canada has been studied.

Very briefly: the first sample of Arcolor 2165, shipped on October 17, 1929, gave promising results, when mixed with shellac, as a base for phonograph records. Subsequent samples of Arcolor 2165 from different batches, — and in fact, one sample from what we have reason to believe is the same batch as that from which the first sample was sent, — have all given unsatisfactory results. Attempts to find another Arcolor which would prove satisfactory have all failed. The men in charge of the work reports that they were unable to determine which of the subsequent samples of Arcolors were most nearly satisfactory, as attempts to check their results gave contradictory results.

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just after the compounder had moved to the new

The problem appears to the writer to be simply a servicing problem. The Victor Company is unable to reproduce their own satisfactory, or nearly satisfactory, results with any recent shipments of Arcolor, and it appears that it is our responsibility to help them find out what has gone wrong, and to make suggestions to correct it. It is recommended that after proper paving of the way, a research man be sent to discuss the problem with Irany and, if possible, to work with the problem there until a solution of the difficulty is found or until a clear conception is obtained of what line of attack we may pursue in the Anniston laboratory.

INTRODUCTION:

The first contact with the Victor Talking Machine Company of Canada consisted in a letter from their Purchasing Agent, dated October 9, 1929, asking for quotations on and samples of Arcolors 1235, 2165 and 4265. One pound each of Arcolor 2165, Arcolor 1234 and Arcolor 4465 were shipped.

On November 25, 1929, Mr. E. P. Irany, of their Chemical Department, wrote that Arcolor 2165 had given quite promising results and that they were willing to do more thorough work towards

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No. 3 - R. W. Coward (C.I.F.)
No. 4 - F. A. Lidbury
No. 5 - L. W. Hutchins
No. 6 -
No. 7 -
No. 8 -

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same letter, Irany suggested cooperative experimenting, involving the preparation by us of special batches of Arcelors, and the testing by them of these batches. "In this way", he states, "it is quite likely that we may succeed to develop something useful". Irany was guarded in his statements regarding eventual use of our Arcelor, although the general tone of the letter was encouraging.

OPERATION:

The method of cooperation pursued since the first letter from Irany has been essentially that requested by Irany. According to the Anniston files, the following have been shipped at the request of either Irany or C. H. Penning:-

<u>Arcelor No.</u>	<u>Weight.</u> <u>- Lbs.</u>	<u>Date of Shipment.</u>
1254	1	Not given on Request Sheet.
2165	1	October 17, 1929.
4465	1	Not given on Request Sheet.
1253	3	December 10, 1929.
4465	3	" " "
2165	3	" " "
5165	3	" " "
2165	100	February 4, 1930.
Diphenyl	1	" 8, "
4065	10	" 8, " (?)
D.H.B.	1	March 3, 1930.
1264	5	" 15, "
2165-1A	2	" 19, "
2165-2A	2	" " "
1269	1	April 1, "
2165	2	" 3, "
(benzene insoluble free)		

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RESEARCH LABORATORY

Swann Research Incorporated

Anniston, Alabama.

REVIEW OF AROCLOR SITUATION FROM STAND-POINT OF USES.

By
H. J. Krass

December 31, 1930.

I N D E X

Aroclors -- Review of Aroclor Situation from
Stand-point of Uses.

Eight copies were made of this report and
distributed as follows:-

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No. 2 - J. N. Carothers
No. 3 - R. W. Coward (C.I.V.)
No. 4 - F. A. Lidbury
No. 5 - L. W. Hutchins
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Classification of Uses of the Aroclors

The classification employed in other reports has been adopted.

They are as follows:--

Varnish

Lacquers

Waterproof Adhesives

Impregnation:-- Fireproofing

Fibre and Transparent Paper

Weather, Moisture and Waterproofing.

Printing Inks

Liquid Dielectrics

Solid Dielectrics

Other Electrical Insulation Problems

Lubricants

Chewing Gum Base

Rubber Compounding

Protective Coatings

Can Sealing

Sealing Wax

Plastics and Plasticizers

Aroclors for Heat Transfer

A considerable number of contacts have been made for which the use is unknown.

The purpose of this review is to examine the outstanding uses to which the Aroclors have been put or are being proposed. In a great many cases little trustworthy information has been obtained from

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the users themselves. A few instances, only, are outstanding exceptions.

Where a sufficiently large number of users have been interested in a single problem or application and there seemed to be justifiable optimism regarding the application of Aroclors, the Research Laboratory has undertaken work on these problems. In this way information has been obtained which should further the commercial introduction of the Aroclors into these new fields. This program will be continued in the future.

In compiling this report the contents of the earlier reports on this subject have been completely revised and combined in this one report and the information brought up to date as of December 31, 1930.

VARNISH

1. What we know about Aroclor in varnish.

(a) H. A. Gardner Paint & Varnish Institute, Wash., D. C.

A report of G's work on diphenyl resin appeared in Circular No. 364 dated April, 1930 of the American Paint & Varnish Mfg's Association. This report points out: 1. Varnishes may be made having a drying time of 4 hours. 2. Some of the varnishes are of very light color. 3. Bleaching action on tung oil noted. 4. Some have been fairly durable in accelerated test and resistant to water. 5. Information as to durability out of doors lacking but will be made this summer. Keeping qualities also lacking. 6. Coatings are alkali resistant and to some extent flame resistant. 7. The harder varities should produce spirit resistant finishes.

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SHERWIN WILLIAMS R.L.J. June 18, 1930.

They found that Aroclor 1254 gave a very flexible film and an "extremely light varnish" lighter than the S-W varnish of the same class.

Specific information contained in Report of visit dated June 18, 1930 on following subjects:

(a) Types of Varnishes for various purposes such as furniture or rubbing, outside uses, auto, electrical such as for varnished cloth and enamelled wire and fire proof varnishes.

(b) Tests for Varnishes. Accelerated Weathering Test. Outside tests and how made influence of various factors such as order of sun and rain. Tests for water proofness, flexibility, gas test, alkali resistance.

(c) Standard Varnishes, for outside use are Valspar or S-W Rex Spar. They also will furnish standard furniture or rubbing varnishes. Electrical varnishes from G.E. Co. No standard fire proofing varnish.

(d) Equipment for laboratory varnish making.

(e) Service of other Rosin Manufacturers. This service consists chiefly in supplying the trade with information as to exactly how to make up varnishes or lacquers from the resin being introduced. Precautions are especially important. An example of how the Bakelite Corp. is attempting to introduce a new resin is given.

The following laboratory reports supplement our knowledge on Aroclors in varnish:

Solubility of Aroclor 4455 in various solvents

June 12-18, 1930.

Refractive Indices of the Aroclors

March 29, 1930.

Report of Call on W.B. Burnett of the Pine Institute of America - Mellon Institute, June 17, 1930.

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Fire-proof Vernishes -- have little information on this type.

Testing of Vernishes -- See Gardner's methods and also the A.S.T.M. tests. Methods of conducting the outside weathering test, water-proofness, flexibility, gas test, and alkali resistance.

Standard Vernishes for Comparison. -- Use Valspar or S-W Rax Spar.

(b) Competitive Materials. Natural varnish gum at 6-10% per lb. ester gum, 10-12%. Amberole type 18-20%.

Our Prices -- 30-40 cents (3 - 3 - 30)

Also note that density of the Aroclors is high, natural resins low.

2. Concerns that have shown active interest.

A. Sherwin Williams Co., Varnish Research

a. What we learned from contacts with them.

Is interested in Gardner's later formula using the viscous Aroclor. They now have both accelerated and long time tests under way. Was interested in our ability to produce large quantities of 1254. They have doubts as to our ability to produce.

b. Samples and Sales.

May 29-29 4 oz. 47 PC (a). 1002 PCR, 1001 - 2PCR.

Nov. 12-29 8 oz. 4465

Mar. 17-50 10 lbs. 4465 - 1254

Apr. 17-30 100 lbs. 1254

May 14 2000 lbs. 1254

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May 16 1 gal. 1242, 10 lbs. 1263, 2565, 4465.

June 11 100 lbs. 4465

July 14, 5 lbs. 1268 new and 4465 new.

Oct. 21 10 lbs. 1268 and 4465

c. Contacts by person and dates.

CHP Feb. 3 - 1930.

B.L.J. Atlanta, April 10, 1930.

C.H.P. April 30, 1930.

d. Conclusion as to future business, size, etc.

They say business amounting to 1 car per month awaiting our ability to produce without delays although no definite assurance of future business has been given. Sept. 10, in answer to Swann's letter, Ware wrote they had made some good varnishes and enamels but certain use problems remain to be worked out. Research hindered by depression.

B. Mubain Paint and Varnish Co., Chicago, Ill.

Subsidiary of the Glidden Co., who do the research.

Want varnish formulas.

Samples: 1001 - 2PG

Contacts: C.H.P. Sept. 16-29 G.B.C. Aug. 29, 1929.

C. Murphy Varnish Co., Newark, N.J. and Chicago, Ill.

Want a retarder for a new combination filler, stain and varnish at Chicago.

Samples: 1219 to Chicago; Mar. 1, 1 lb. 1254; Mar. 21, 1 gal 1262;

Sept. 20, 10 lbs. 4465 to Newark.

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