REPORT NO. 2215

FINAL REPORT
ON

AROCLOR DATA BOOK

Job No. 171-451

File No. 141-27.1

RESEARCH DEPARTMENT - PHOSPHATE DIVISION

Anniston, Alabama
****

Report Submitted - April 27, 1948

Chemists: A.M. Ellenburg
R.R. Knight

Prepared by: R.R. Knight

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RESEARCH DEPARTMENT - PHOSPHATE DIVISION

MONSANTO CHEMICAL COMPANY

Anniston, Alabama

AROCLOR DATA BOOK

General information on properties of Aroclors, Aroclor process data, uses of Aroclors, and physiological effects of Aroclors.

FOREWORD

As a means of presenting the available data on Aroclors to the interested personnel within the Monsanto organization, this looseleaf notebook is being compiled.

Most of the data has resulted from work carried out within our own organization. Literature references, however, will be cited in all cases throughout the compilation in order to allow more detailed information to be obtained by the user.

The following detailed outline is for facilitating the location of data in the book and to assist in properly inserting new data sheets. Each Aroclor is to be given a series number for location under the general headings. This scheme at present is as follows:

Aroclor 1221	-	100 series
Aroclor 1232	-	200 series
Aroclor 1242	-	300 series
Aroclor 1248	-	400 series
Aroclor 1254	-	500 series
Aroclor 1260	-	600 series
Aroclor 1262	-	700 series
Aroclor 1268	-	800 series
Aroclor 1270	-	900 series
Aroclor 1271	-	1000 series
Other Diphenyl Aroclors	-	1100 series
Aroclor 4465	-	1500 series
Other High Boiler Aroclors	-	2100 series
Aroclor 5442	-	2400 series
Aroclor 5460	-	2500 series
Aroclor 5465	-	2600 series
Aroclor 5468	-	2700 series
Aroclor 2565	-	3000 series
Related Compounds	-	4000 series

DSW 331653

The page pertaining to the solubility of Aroclor 4465 in various solvents would be of this type "IAj - 1500". In cases where the information for all Aroclors can be compiled on one sheet, such as refractive indices, the page will be inserted under the general heading and will bear only the number for the first Aroclor, for example, IAh - 100.

I. GENERAL PROPERTIES OF AROCLORS

A. Physical Properties

(a) General Physical Constants

1. Formula and molecular weight

2. Specifications for Manufacture

(b) Density and Specific Gravity

(c) Cubical Coefficients of Expansion

(d) Vapor Pressure and Rate of Evaporation

(e) Specific Heat and Heat Capacity

(f) Thermal Conductivity

(g) Viscosity

(h) Refractive Index

(i) Heats of Vaporization - Other Thermodynamic Properties

(j) Solubilities

(k) Flash and Flame Points

(l) Miscellaneous

B. Electrical Properties

(a) Dielectric Constants

(b) Power Factors

(c) Dielectric Strength

(d) Volume Resistivity

(e) Dipole Moments

(f) Miscellaneous

DSW 331654

II. METHODS OF MANUFACTURE

A. Monsanto Process

1. Raw Materials

2. Chlorination

3. Distillation

4. Storage and Shipping

B. German Process

C. Other Processes

III. USES OF AROCLORS

A. Electrical Field

1. Transformers

2. Capacitors

3. Coating for Wire

4. Other uses

B. Varnishes

C. Plasticizers

D. Hydraulic Fluid

E. Fire Retardants

F. Heating medium

G. Miscellaneous

H. Suggestions for new Uses

IV. PHYSIOLOGICAL EFFECTS

1. Skin Tests

2. Systematic Tests

PATENTS USING AROCLORS

The book is subject to revision and changes as various data are located. Graphs and diagrams will be inserted when possible and new graphs drawn as information is compiled.

No indices of sections is planned at this time, but later revisions may include such pages if the volume of information warrants it.

Contributions of new and supplementary data are earnestly solicited. Any discrepancies in the existing data or advice as to a better means of presenting the information will be greatfully acknowledged. Address all correspondence to the group leader in charge of the research on Aroclors at Phosphate Division - Research Department, Anniston, Alabama.

R. R. Knight

R. R. Knight

A. M. Ellenburg

A. M. Ellenburg

rs
12/2/47

STANDARD SPECIFICATION OF

MONSANTO CHEMICAL COMPANY
PHOSPHATE DIVISION
ANNISTON, ALABAMA

PRODUCT: Aroclor 1221 **CODE NO.:** 1040-225-75-09

GRADE: Regular **DATE AUTHORIZED:** December 4, 1946

PROD. DEPT. NO.: _____ **SUPERSEDES:** New Tentative

APPROVED BY (Initials) R.L.J.; 12-17-46 A.M.E.; 12-19-46 J.F.R.; 12-19-46 F.A.B. 12-19-46

	Control Specification	Consumer Specification
Crude Aroclor 1121:		
Sp. Gr. at 65°C.	1.150-1.160	
Acidity, mg. NaOH/gm.	≤0.5	
Aroclor 1221:		
Color	40 APHA max.	
Acidity, mg. NaOH/gm.	0.01 max.	
Sp. Gr. at 65/15.5°C.	1.145-1.155	
Chlorine content	20.5-21.5%	
Viscosity at 100°F.	38-41 SUS	

NOTES:

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STANDARD SPECIFICATION OF

MONSANTO CHEMICAL COMPANY
PHOSPHATE DIVISION
ANNISTON, ALABAMA

PRODUCT: Aroclor 1232 **CODE NO.:** 1040-230-75-09

GRADE: Regular **DATE AUTHORIZED:** December 4, 1946

PROD. DEPT. NO.: _____ **SUPERSEDES:** New-Tentative

APPROVED BY (Initials) R.L.J.; 12-17-46 A.M.E.; 12-18-46 J.F.R.; 12-19-46 F.A.B. 12-19-46

	Control Specification	Consumer Specification
Crude Aroclor 1132:		
Sp. Gr. at 65/15.5°C.	1.240-1.245	
Acidity, mg. NaOH/gm.	≤0.5	
Aroclor 1232:		
Color	50 APHA max.	
Acidity, mg. NaOH/gm.	.01 max.	
Sp. Gr. at 65/15.5°C.	1.235-1.240	
Chlorine content	31.5-32.5	
Viscosity at 100°F.	46-49 SUS	

NOTES:

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STANDARD SPECIFICATION

OF

MONSANTO CHEMICAL COMPANY

Product: Chlorinated Diphenyl, distilled Code No. 1040-240-75-09

Grade: Aroclor 1242 Date Authorized June 21, 1940

Supersedes Specification Dated July 3, 1934

	Tolerable Limits	Typical Value
Sp. Gr. at 65/15.5°C.	1.338 to 1.348	
Color, N.P.A.	0.5 Maximum	
Acidity, Mgm NaOH/gm.	.01 Maximum	
Viscosity at 54.4°C.	47 to 50 Seconds Saybolt Universal	

Approved by A. B. Gerber
Chief Chemist

Approved by Robert S. Weatherly
Sales Manager

Approved by Edw. A. O'Neal, Jr.
Works Manager

Authorized by J. N. Carothers
Chemical Director

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STANDARD SPECIFICATION

OF

MONSANTO CHEMICAL COMPANY

Product: Chlorinated Diphenyl, distilled Code No. 1040-260-75-09

Grade: Aroclor 1248 Date Authorized June 21, 1940

Supersedes Specification Dated July 3, 1934

	Tolerable Limits	Typical Value
Sp. Gr. at 65/15.5°C.	1.404 to 1.414	
Color, N.P.A.	0.5 Maximum	
Acidity, Mgm NaOH/gm	.01 Maximum	
Viscosity at 54.4°C.	69 to 76 Seconds Saybolt Universal	

Aroclor 1262 Code No. 1040-310-75-09

Sp. Gr. at 90/15.5°C.	1.572-1.583
Color, N.P.A.	1.0 Maximum
Acidity, Mgm NaOH/gm.	.01 Maximum
Viscosity at 98.9°C.	88-100 SUS

Approved by A. B. Gerber
Chief Chemist

Approved by Robert S. Weatherly
Sales Manager

Approved by Edw. A. O'Neal, Jr.
Works Manager

Authorized by J. N. Carothers
Chemical Director

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STANDARD SPECIFICATION

OF

MONSANTO CHEMICAL COMPANY

Product: Aroclor 1254 Code No. 1040-280-75-09

Grade: Dielectric Date Authorized 10/3/41

Supersedes: 6/21/40

Color, APHA Scale	100 Max.
Condition	Clear
Specific Gravity at 65/15.5°C	1.495-1.505
Acidity, Mgm. NaOH/gm.	.01
Inorganic Chlorides, ppm.	0.10 Max.
Saybolt Viscosity at 98.9°C, sec.	44.5-47.5
Dielectric Constant at 100°C	4.15-4.35
Resistivity at 100°C, ohm-cm. at 500 volts	Above 500 X 10^9
Refractive Index at 25°C	1.6370-1.6390
Distilling Range, Observed, 10%	350-355°C
Observed, 50%	355-362°C
Observed, 90%	362-375°C
Pour Point	8 to 12
Water, ppm.	35 Max.
Evaporation, 6 Hrs. at 100°C	0.4%
Corrosion Test-Change in weight	0.0%
Acidity _____ after test	0.01
Inorganic Chlorides _____ after test	0.10 Max.
Condition _____ after test	Clear
Color _____ after test	150 Max.

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STANDARD SPECIFICATION

OF

MONSANTO CHEMICAL COMPANY

Product: Aroclor 1260 Code No. 1040-290-75-09

Grade: Dielectric Date Authorized 11/18/41

Supersedes: 1/30/36

Color, APHA Scale	100 Max.
Condition	Clear
Specific Gravity at 90/15.5°C	1.550-1.560
Acidity, Mgm. NaOH/gm.	.01
Inorganic Chlorides, ppm.	0.10 Max.
Saybolt Viscosity at 98.9°C, sec.	75-80
Dielectric Constant at 100°C	3.6-3.8
Resistivity at 100°C, ohm-cm. at 500 volts	Above 500 X 10^9
Refractive Index at 25°C	1.6455-1.6465
Distilling Range, Observed, 10%	370-377°C
Observed, 50%	377-385°C
Observed, 90%	385-400°C
Pour Point	26 - 34
Water, ppm.	35 Max.
Evaporation, 6 Hrs. at 100°C	0.2 %
Corrosion Test-Change in weight	0.0%
Acidity _____ after test	0.01
Inorganic Chlorides _____ after test	0.10 Max.
Condition _____ after test	Clear
Color _____ after test	150 Max.

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DSW 331662

SPECIFICATIONS FOR SOLID DISTILLED AROCLORS

PRODUCT	APPEARANCE & COLOR	HOLD POINT	ACID NO. Mg.NaOH/gm	COLOR 5% TOLUENE	MELTING POINT	TOTAL CHLORINE
1268	White to yellow crystalline powder	135-160°C.	0.05 Max.			
1269	White to grey or light yellow crystalline powder	225-255°C.				
1270	White crystalline powder	285-300°C		80 APHA Max.		69.5 - 70.5%
1271	Practically white light fluffy powder	-		80 APHA Max.	304°C. Min.	

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STANDARD SPECIFICATION OF

MONSANTO CHEMICAL COMPANY
PHOSPHATE DIVISION
ANNISTON, ALABAMA

PRODUCT: Arocolor 4465 (Regular) **CODE NO.:** 1040-430-75-09

GRADE: _____ **DATE AUTHORIZED:** October 4, 1944

PROD. DEPT. NO.: _____ **SUPERSEDES:** June 3, 1944

APPROVED BY (Initials) R.L.J.; 9-29-44 H.F.T.; 9-29-44 P.L.; 10-2-44 R.G.T.; 10-2-44 F.A.B. 10-4-44

	Control Specification	Consumer Specification
Appearance :	clear, light yellow, brittle resin	
Color, N.P.A.*	2.0 max.	
Softening Point (A.S.T.M.)	60 - 66°C.	
Acid Number (MgNaOH/gm.)	0 - .035	
Crystallinity	No. spec.	

* Raising color limit to 2.0 maximum is recommended because inspection records show that all lots produced in 1944 have had a color of 1.5%.

NOTES:

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STANDARD SPECIFICATION

OF

MONSANTO CHEMICAL COMPANY

Product: Chlorinated High Boiler Code No. 1040-480-75-09

Grade: Aroclor 5460 Date Authorized May 10, 1940

Supersedes Specification Dated December 23, 1932

	Tolerable Limits	Typical Value
Appearance	Clear, light yellow, brittle resin	
Color, N.P.A.	2.0 maximum	
Crystallinity Test	To pass test	
Softening Point, ASTM	100 - 105.5°C.	
Acid Number Mgm NaOH/gm.	0 - .05	
Chlorine	59.0-80.6%	

Approved by A. B. Gerber
Chief Chemist

Approved by Robert S. Weatherly, 5/6/40
Sales Manager

Approved by Edw. A. O'Neal, Jr.
Works Manager

Authorized by J. N. Carothers, 5/10/40
Chemical Director

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Monsanto Chemical Company
Anniston, Alabama

Aroclor Test Methods and Designations

Specific Gravity of Aroclors	14-10-48
Total Chlorine in Aroclors	14-13-48
Softening Point of Solid Aroclors	14-17-48
Determination of Iron in Aroclor	14-21-48
Flash and Flame Points	14-24-48
Viscosity of Liquid Aroclors	14-29-48
Distillation Range of Aroclors	14-31-48
Evaporation Test of Liquid Aroclors	14-32-48
Refractive Index of Liquid Aroclors	14-34-48
Resistivity of Liquid Aroclors	14-35-48
Dielectric Constant of Liquid Aroclors	14-36-48
Acid Number of Liquid Aroclor	14-42-48
Color of Aroclor - NPA Scale	14-43-48
Color of Aroclor - APHA Scale	14-44-48
Acid Number of Solid Aroclors	14-46-48
Pour Point of Liquid Aroclors	14-47-48
Inorganic Chlorides in Aroclors	14-48-48
Water Content of Liquid Aroclors	14-53-48

DSW 331666

From Letter to W.C.M./M.T.Bolmer (Halowax Products Div., Union Carbide and Carbon Corp., 30 E. Forty-Second St., N.Y. 17, N.Y.). Letter dated Feb. 27, 1948.

PROPERTIES OF BASIC "HALOWAX" PRODUCTS

MATERIAL NUMBER	No. 1000	No. 1001	No. 1013	No. 1014
Colors	White to Pale Straw	White to Pale Yellow	Pale Yellow	Pale Yellow
Flow Point (Mod. ASTM Softening Point) deg. F.	Liquid	194-201	247-252	277-283
Specific Gravity at 77 deg. F.	1.19 - 1.25	1.53-1.59	1.65-1.71	1.75-1.81
Penetration (Mod. ASTM) 200-g. load at 77 deg. F.	—	10-15	5-8	5-8
Viscosity (Saybolt Universal Seconds)	33-37 at 77 deg. F.	33-37 at 266 deg. F.	31-35 at 266 deg.F.	33-37 at 302 deg.F.
Distillation Range (ASTM) deg. F.	480-590	600-650	615-655	680-730
Flash Point (ASTM Cleveland Open Cup) deg. F.	203	284	356	392
Fire Point (ASTM Cleveland Open Cup) deg. F.	338	None to boiling	None to boiling	None to boiling
Power Factor at 1,000 cycles at 10^6 cycles	— —	Less than 0.2% 0.43%	Less than 0.2% 0.70%	Less than 0.2% 0.70%
Dielectric Constant at 1,000 cycles at 10^6 cycles	— —	5-6 5-6	4-5 4-5	4-5 4-5
D. C. Resistivity (meg. cm.) at 30 deg. C.	—	Over 10^7 - 10^8	Over 10^7 - 10^8	Over 10^7-10^8

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DSW 331667

I-A-B-4000

From - Halowax Products Division
Union Carbide and Carbon Corporation
30 East 42nd Street
New York 17, New York

Letter - W.C.M./J.M. Cole, Tech. Rep.
2/16/48
Zyrox 3009 (11-313)

GENERAL INFORMATION

Color	- Brown
Sp. Gravity	- 1.40-1.45 at 25°C.
Softening Pt.	- 79-83°C.
Stormer Viscosity	- 59-89 at 130°C.
Saybolt Furol Viscosity	- 79-180 at 130°C.
Penetration at 50°C.	- Approx. 15 (200 gm. load)
Penetration at 25°C.	- Less than 3 (200 gm. load)
Flash Pt.	- Approx. 590°F.
Fire Pt.	- None to 600°F.
Combustibility	- Will not support combustion
Fracture	- Conchoidal
Acid Resistance	- Excellent
Alkali Resistance	- Excellent
Volatility	- Average for 24 hours at 130°C. Less than 0.10 mg./sq.cm./hr.
P.F. at 25°C. (1000 cycles)	- Approx. .001
Diel. Constant at 25°C. (1000 cycles)	- Approx. 2.96
DC Resistivity at 25°C.	- Over 10^8 meg. cms.

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DSW 331668

From - Halowax Products Division
Union Carbide and Carbon Corporation
30 East 42nd Street
New York 17, New York

Letter - W.C.M./J.M. Cole, Tech. Rep.
2/16/48
Zyrox 3007 (11-308)

GENERAL INFORMATION

Color	- Brown
Sp. Gravity	- 1.29 - 1.32 at 25 deg. C.
Softening Pt.	- 65 - 70 deg. C.
Stormer Viscosity	- 20-27 at 130 deg. C.
Saybolt Furol Viscosity	- 20-30 at 130 deg. C.
Penetration at 50 deg. C.	- Approx. 75 (50 g. load)
Penetration at 25 deg. C.	- Less than 3 (200 g. load)
Flash Pt.	- Approx. 530 deg. F.
Fire Pt.	- None to 600 deg. F.
Combustibility	- Will not support combustion
Fracture	- Conchoidal
Acid Resistance	- Excellent
Alkali Resistance	- Excellent
Volatility	- Average for 24 hrs. at 130 deg. C. 0.20 mg./sq.cm./hr.
P.F. at 25 deg. C. (1000 cycles)	- Approx. .004
Diel. Constant at 25 deg. C. (1000 cycles)	- Approx. 3.0
DC Resistivity at 25 deg. C.	- Over 10^8 meg. cms.

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DSW 331669

From: B.I.O.S. Final Rept. No. 893
Item Nos. 1, 7, 22, & 31
Pages 108 - 109

APPENDIX II. PHYSICAL AND CHEMICAL PROPERTIES OF CLOPHEN OILS

(as quoted in I. G. Farben's sales sheets)

Property	Clophen A30	Clophen A40	Clophen A50	Clophen A60	Clophen A70	Clophen A80
Form	Liquid	Liquid	Liquid	Liquid	Firm	Firm, crystalline
Colour	Nearly colourless	Nearly colourless	Nearly colourless	Nearly colourless	Nearly colourless	White
Specific Gravity at 20°C	1.35	1.48	1.55	1.68	1.7	1.72 - 1.77
Solidifying Point	-18°C	-5°C	+12°C	+30°C	+50°C	>160°C
Flash Point*	166° (Pensky - Martens)	193°	222° In an open crucible	236°	240° (Pensky - Martens)	290° or over
Shrinkage: 100° - 20°C	6%	6%	6%	6%	5 - 6%	-
50° - 20°C	3%	3%	3%	3%	3%	-
Viscosity at 100°C	1.1°E	1.2°E	1.5°E	1.9°E	4°E	-
Boiling Point Limit at 12mm. of Mercury	165 - 195°	180 - 215°	190 - 230°	195 - 250°	200 - 255°	210 - 260°
Loss during Evaporation (after 6 hours at 100°C)	0.05%	0.03%	0.02%	<0.01%	0.01%	0.0 (2 hrs. at 125°)
Volatility at 20°C (1mm. of Mercury for 100 hours)	<0.009%	<0.009%	<0.009%	<0.009%	<0.009%	<0.009%
Heat Conductivity at 40°C (in Calories per metre per hour per °C).	0.095	0.091	0.087	0.086	-	-
Saponification No.	0.0	0.0	0.0	0.0	0.0	0.0
Ash	V. small	V. small	V. small	V. small	V. small	V. small
Permittivity at 20°C	6.0	5.4	5.0	4 - 4.5	3.0	3.0
Power Factor	<0.001	<0.001	<0.001	<0.001	<0.001	-
Breakdown Strength	200kV/cm.	200kV/cm.	>200kV/cm.	>200kV/cm.	-	-
Insulation Resistance in Ohm.cm.	1×10^{13}	1×10^{13}	1×10^{14}	$> 1 \times 10^{14}$	$> 1 \times 10^{14}$	$> 1 \times 10^{14}$

* Clophens are not combustible and do not support combustion of materials into which they are impregnated.

I-A-B-10

DSW 331670

STLCOPCB4078245

TABLE I

	Hydrocarbon compositions		Properties of dielectric			
	Diphenyl	Distilled high boiling compounds	Chlorine content	Viscosity sec. Saybolt at 210° F.	Specific gravity at 65°/65° C.	Dielectric constant K at 100° C. (1,000 cycles)
	Percent	Percent	Percent			
1 - - -	0	100	23.9	63	1.251	4.35
2 - - -	0	100	40.3	290	1.405	4.98
3 - - -	80	20	41.2	35.7	1.352	4.87
4 - - -	80	20	52.2	47.5	1.490	4.56
5 - - -	50	50	42.0	48.8	1.383	4.84
6 - - -	50	50	36.4	40.8	1.324	4.89
			Properties of chlorinated diphenyl			
7 - - -	100	0	54.0	46.0	1.523	4.30
8 - - -	100	0	42.0	34.0	1.380	4.80

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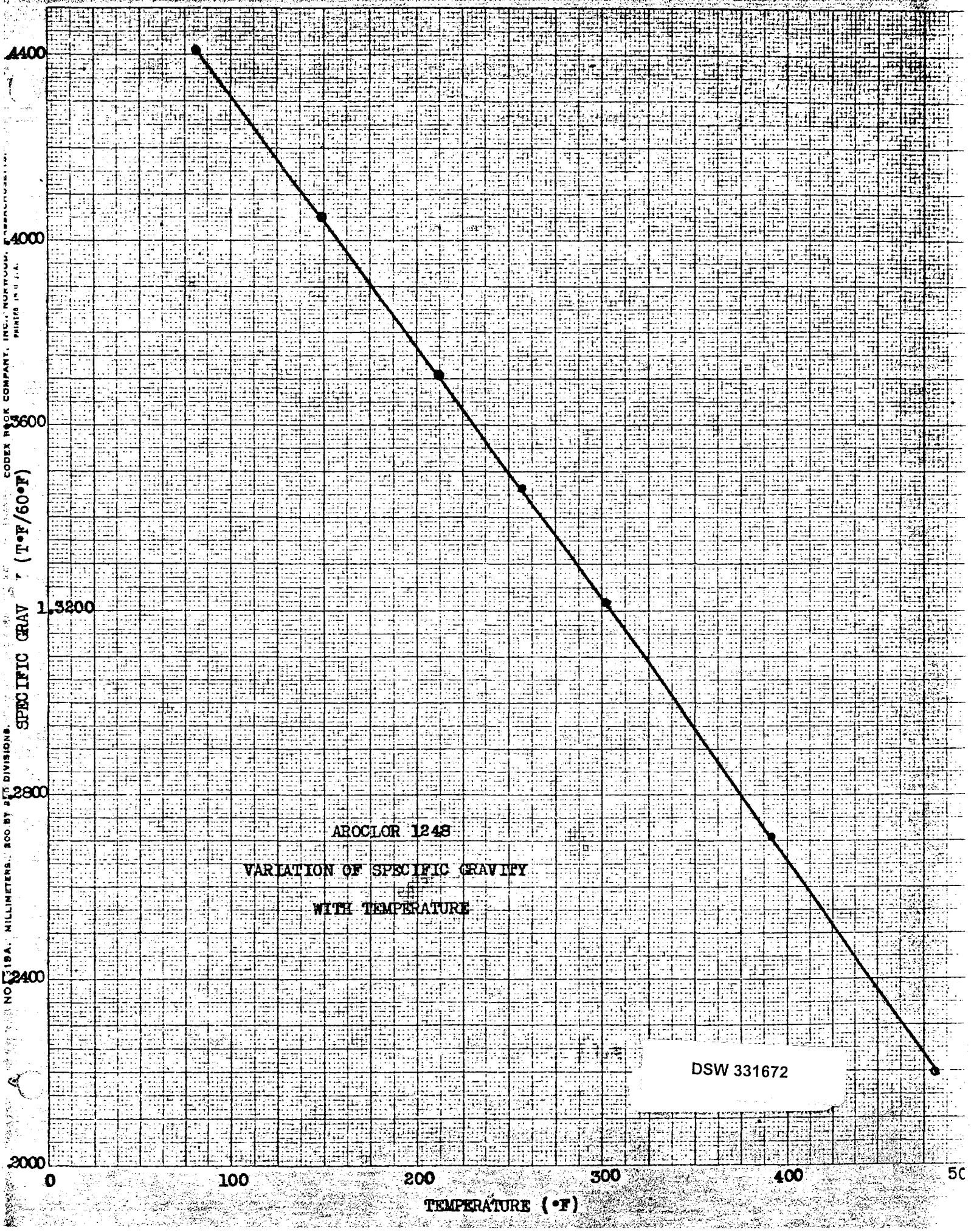

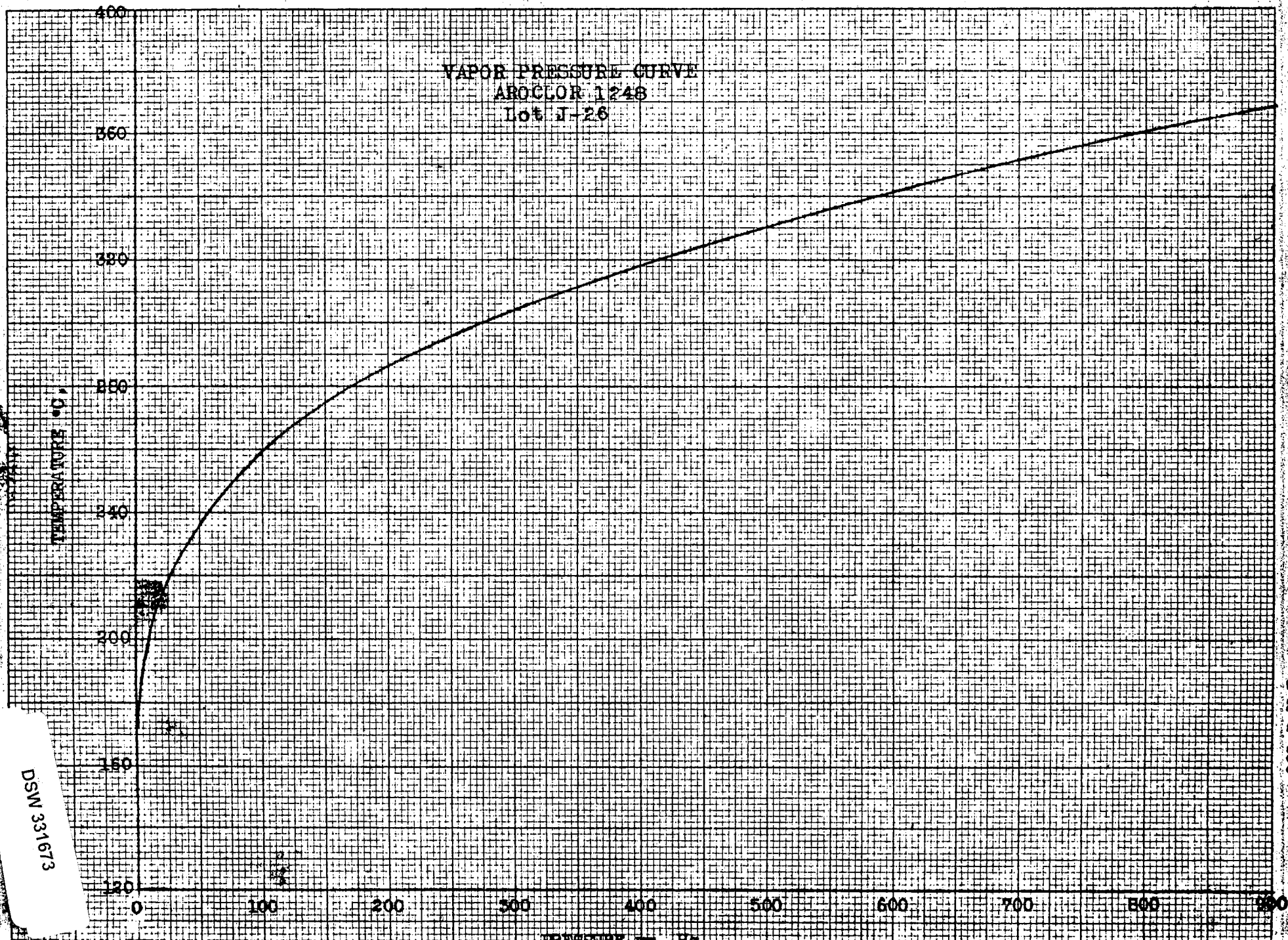

VAPOR PRESSURE CURVE
ARCTOR 1234

P. in. Hg.

800
700
600
500
400
300
200
100
0

TEMPERATURE °F.

400
360
320
280
240
200

Stability of Grade P Hycar and Teflon in Aroclor 1254 at 130°C and 45°C

Short Form Report No. 2179

File No. 141-27.1

December 29, 1947

Notebook references: Smith 45137

Grade P Hycar and Teflon Immersed in Aroclor 1254

Material	Temp.	Hours	% Gain in Wt.	% Gain in Thickness
Grade P Hycar	130°C	285	68.6	26
Grade P Hycar	45°C	285	26.3	6
Teflon	130°C	285	Negligible	Negligible
Teflon	45°C	285	Negligible	Negligible

rs
1/14/48

DSW 331675

I-A-1 - 500

From - Memo P.G.B./R.L.J. 3/22/48

Aerovox Corporation
New Bedford, Massachusetts

Determining Fluorescence in Aroclor

"The ultraviolet light we use for determining fluorescence in Aroclor is manufactured by George W. Gates and Company, Franklin Square, Long Island, New York. It is a CH 4, 100 Watt, G. E. Mazda Lamp with filter.

"Our procedure is to pour a small amount of Aroclor into a 5" watch glass or a 5" Pyrex evaporating dish and observe for the degree of fluorescence under the CH 4 lamp, preferably in a dark room. If the sample has been contaminated with Mineral Oil it will show up as a streaky or milky fluorescence as compared to pure Aroclor. The fluorescence around the meniscus of pure Aroclor is not to be confused with the fluorescence resulting from contamination.

"This test is qualitative and, as such, is based on the experience of the person performing it. We keep a standard sample for comparison. If there are any further questions please feel free to contact us".

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4/2/48

DSW 331676

IB - 100

December 2, 1947

ELECTRICAL DATA ON AROCLORS

Introduction

Time has not permitted us to carry out electrical measurements on all of the Aroclor samples sent by your laboratory and as listed in your letter to Dr. C. K. Bump of June 27, 1947. It was believed that dielectric constant, loss factor, and direct current resistivity measurements over a temperature range for a low chlorinated and highly chlorinated biphenyl; also one sample each of the terphenyl and the mixed biphenyl and terphenyl series would represent the electrical behavior of Aroclors.

The trend of the electrical properties of the samples not measured can be estimated; i.e. as the viscosity of the Aroclors of a particular series increases, the loss factor will decrease and the loss factor maximum (if within the temperature range measured) will be shifted to higher temperatures. An increase in viscosity will increase the resistivity of the Aroclor. The dielectric constant of a material is a measure of its polarizability. Therefore, any change in the structure of the molecule which will increase its polarizability will cause an increase in dielectric constant.

Accuracy of Data

The accuracy of the alternating current data is dependent on the dissipation factor of the dielectric at a particular frequency and a particular temperature. The dielectric constant values are accurate within .2% provided the dissipation factor is less than 10%. The error is greater with increasing dissipation factor. The accuracy of loss factor (dielectric constant X dissipation factor) is also dependent on dissipation factor. Aroclor 5442 was the only sample which had a dissipation factor greater than 10% and that occurred at the following conditions:

41°C, 500 cycles
41°C, 750 cycles
35°C, 500 cycles

It will be noted that the dielectric constants extrapolated to 100°C. check satisfactorily with the values listed in your letter to Dr. Bump.

The direct current resistivity values have a 30% error for the greatest resistance readings on our instrument. For lower resistance values the error decreases. Aroclors 1221, 1254 showed an increase in resistance with time which is indicative of polarization effects. For these materials then, resistance was read after the voltage was applied and then at the end of one minute - an empirical method used for taking a readable value. Aroclors 4465 and 5442 did not show noticeable pola-

rization effects, even at the high temperatures.

Data

The dielectric constant of Aroclor 1221 and 1254 is independent of frequency. The dielectric constant of Aroclor 4465 and 5442 is also independent of frequency at the high temperatures.

The liquid-like properties of 4465 and 5442 disappear at the lower temperatures where a dispersion region becomes evident. It will be noticed that the dielectric constant curves (at the lower temperatures) have the same order as those of the corresponding loss factor curves and the inflection regions occur at the loss factor maxima temperatures.

The loss factor curves of 5442 at the higher temperatures show a sharp up-swing, indicative of a large conductance component in the loss factor. Similarly, the loss factor curves of 1221 and 1254 seem to be in the conductance region.

Aroclor 1221 showed an unusual sensitivity in changing loss factor values and as great an insensitivity to change in dielectric constant. It was found that a sample measured initially at room temperature and measured at successively increasing temperatures gave different loss factor values when compared to measurements made with the initial temperature high and then successively lowered. Results showed that raising the temperature of the liquid or allowing the material to stand in the open for long periods always lowered the loss factor, probably volatilizing the more conducting or the high loss substances in the Aroclor. Below 5000 cycles this effect was very noticeable, the differences increasing with decreasing frequency. It may be mentioned here that loss factor measurements at low frequencies might possibly be used as an analytical tool or as a control method of indicating volatile substances contained in the Aroclor 1200 series.

The log resistivity versus 1/T graph is useful as an indication of the change that occurs in resistivity with temperature - or what is related, the change in viscosity with temperature. The actual slopes of 4465 and 5442 cannot be assured since there should have been more measurements made at high temperatures. However these two Aroclors have a much larger viscosity-temperature coefficient than the other two plots.

It is hoped that the enclosed graphs will summarize the dielectric properties of the Aroclors measured. More complete information or any questions concerning the data will be sent on request.

R. Levreault
Monsanto Chemical Company
Plastics Division
Springfield, Massachusetts

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1/15/48

From - B.I.O.S. Final Report No. 893
Item Nos. 1, 7, 22, and 31
Pages 12, 13, 14, 15, 16, & 17

III. CHLORINATED DIPHENYL.

1. General.

Chlorinated diphenyl is manufactured by I.G. Farben under the trade name of Clophen. Some types are liquid and others solid at normal temperatures. They are used as impregnants as alternatives to hydrocarbon oils and waxes. Clophen shares with other chlorinated materials a high permittivity and has the same dangers in handling, due to noxious properties. In addition to the grades suited to capacitor impregnation, a grade known as T64 is made by I.G. Farben, suitable for transformer cooling. The total output of Clophen A50 and A60 for the years 1938 and 1943 was:-

1938	Type	A50	21 tons
	"	A60	132 "
1943	"	A50	170 "
	"	A60	506 "

The cost of the material in Germany is:-

Type A50 1M. 50pfg. to 1M. 60pfg. per Kilogram
" T64 1M. 20pfg. per Kilogram.

The following is a list of the principal prewar consumers of Clophen:-

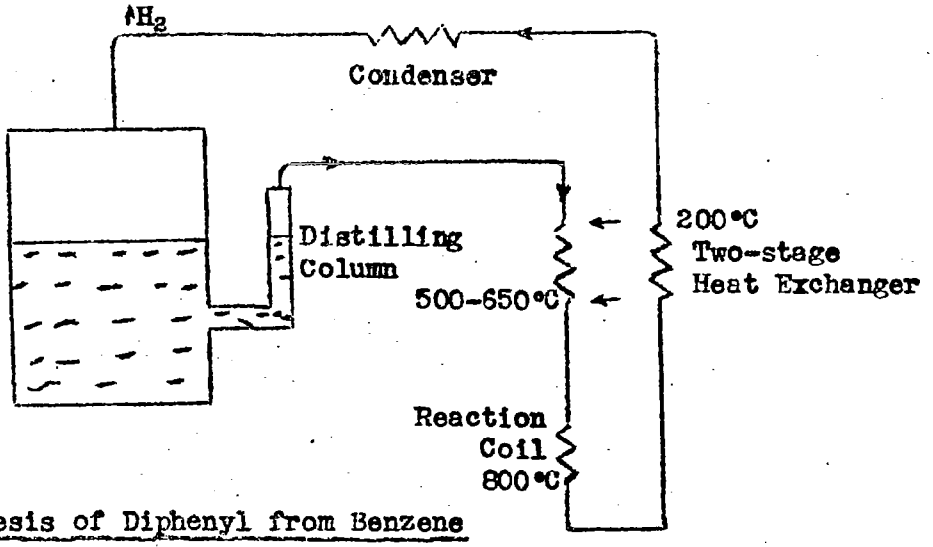
Siemens-Schuckert, Berlin.
A.E.G. (Hydrawerk), Berlin.
Micafil (Brown Boveri), Zurich, Switzerland.
Alsthom, Paris.
Ducati, Milan, Italy.

It was stated that no German capacitor manufacturer uses Clophen for the manufacture of small capacitors for radio and telephone purposes and where these have been used by the German fighting services they were supplied by the Italian firm, Ducati.

2. Manufacture.

(1) The synthesis of diphenyl from benzene.

Diphenyl is synthesised from benzene as shown in the figure, following:-



Synthesis of Diphenyl from Benzene

The synthesis occurs in the vapour phase, at 800°C. and atmospheric pressure, in an electrically heated reaction coil of copper-manganese alloy. No catalyst is required. The apparatus, apart from the reaction coil, is of iron, and operates at the temperatures shown above. About 10% of the benzene vapour is converted into diphenyl in one passage through the reaction coil.

The purity of the benzene is of importance for the electrical quality of the ultimate chlorinated products and a specification is attached overleaf. The benzene used was believed to contain 0.2 to 0.3% of paraffins but little definite information could be obtained regarding any undesirable impurities or the mechanism by which they cause poor electrical performance. Thiophene was stated to be definitely harmful, and vague reference was made to "nitro-compounds", to "aliphatic hydrocarbons" and to "substances which could not be chlorinated". The precise nature of these materials was not known, but their absence was ensured by obtaining benzene always from suppliers whose product was known to be satisfactory.

Analytical Specification for Pure Benzene

Appearance:	Clear and colourless.
Density at 20°C.:	0.876
Distilling range:	First 5% within 0.25°C. 5% - 95% " 0.25°C. 95% - end " 0.25°C.
Solidification point:	Not less than 5°C.
Bromine consumption:	Not more than 0.5 gm. bromine per 100 c.c.
Sulphuric acid test:	Not more than 0.15 gm. $K_2Cr_2O_7$
Carbon disulphide:	Shall be free from CS_2.

DSW 331680

Difficulty in the synthesis was still being experienced, due to rapid corrosion of the copper-manganese reaction coil, operating at 800°C. A small scale experimental apparatus using a fused silica coil had been made, but had not been developed for production use.

In addition to diphenyl, about 5% each of 1-3 and 1-4 terphenyls are produced by the process. These are separated, chlorinated and used in sealing varnishes; present production is about 4 tons per month. No use is made of chlorterphenyls as paper impregnants, since the materials are resinous and tend to crack; moreover the permittivities are lower than those of the chlordiphenyls.

The crude diphenyl is next purified by distillation. During this process the terphenyls remain in the residue. They are extracted in the following manner:-

The residue is dissolved in hot benzene, filtered, and the 1-4 isomer obtained by fractional crystallization. The crude product is distilled in vacue, yielding 1-4 terphenyl with melting point of 208°C. The 1-3 isomer, remaining in solution in benzene, is more difficult to isolate. The solution is clarified with activated charcoal, filtered, and the benzene evaporated. The residue is distilled to yield 1-3 terphenyl with melting point 80-84°C.

(2) Chlorination of diphenyl.

For the chlorination of diphenyl a lead lined vessel of 10,000 litres capacity is used. It is charged with 6000 kg. of diphenyl and 15 kg. of ferric chloride. The mixture is heated to 110°C., agitated and chlorine passed in. The addition of chlorine is continued for 100 hours at a rate of 130-135 kg/hour during which time the temperature is raised gradually from 110°C. to 130°C. The end point of the chlorination is determined by measuring the density of the product.

To remove hydrogen chloride formed in the process, dry air is blown through the liquid for 2 hours and this hydrogen chloride is recovered.

The product is transferred to another vessel and treated with 1/2% of solid sodium hydroxide, heated and agitated. The sodium hydroxide is allowed to settle out and the Clophen drawn off from the top, transferred to another vessel to which Fuller's earth and soda are added and the liquid redistilled. The stills for this process are gas heated.

3. Properties.

(1) Capacitor impregnating grades.

A summary of the physical and chemical properties of the capacitor impregnating grades of Clophen is given in Appendix II. There are six grades of capacitor impregnant, A30, A40, etc. up to A80, the 3, 4, 5, etc. indicating the number of chlorine radicals combined with the diphenyl. Each grade contains a proportion of the adjacent homologues not exceeding 20%. Grade A30 has the highest value of permittivity. Theoretically the highest value obtainable with a chlorinated diphenyl is 9.0, and a grade having a permittivity of 7.0 has been prepared, but is chemically unstable, and very sensitive to moisture and impurities. The most commonly used grades are A50 and A60; A50 is the most stable grade and has the lowest dielectric loss. Grade A60 is considered by I.G. to possess the optimum combination of non-inflammability, stability and viscosity. As with Nibren, it is considered that no additives are required since the substances are stable in use, provided that the capacitors are efficiently sealed against the air. Micafil of Zurich are the only users of A30 and A40.

The following table shows the variation in capacitance and power factor with temperature for a Clophen impregnated paper capacitor.

The impregnant is A50, the dielectric is Schoeller and Hoesch "A" finish rag tissue (density 1.2 - 1.25) with a thickness of 10 u; frequency of measurement is 800 c/s. Power factor shows a maximum value of 0.052 at about +4°C. while capacitance falls off by some 20% at temperatures below this critical value.

Variation in capacitance and power-factor with temperature for a Clophen-impregnated capacitor.

Temperature (°C.)	Relative Capacitance	Power Factor
-60	0.87	0.035
-40	0.91	0.024
-20	0.94	0.013
0	1.02	0.048
+4	1.06	0.052 (max.)
+20	1.10	0.006
+40	1.10	0.005
+60	1.10	0.004
+80	1.11	0.005

No evidence was obtained from I.G. on the use of any type of additive with a view to depressing the temperature at which the change of state occurs.

The method adopted for evaluating the water content in oils such as Clophen is described in Appendix III. It is also applicable to such solids as Nibren.

Engelhardt stated that no source of failure had been experienced during the whole period of production of Clophen, which is about 17 years. Consequently no improvements or modifications had been made or found necessary during this period.

(2) Transformer-cooling grade (Clophen T64).

No detailed information was sought on this material, since it is unsuitable as a capacitor impregnant and is only used as a coolant for transformers and phase shifters. A summary of its physical and chemical properties is given in Appendix IV. It has a low viscosity, is moderately stable under electrical stresses and has the distinct advantage over mineral oils of being non-inflammable.

The principal users of this product are A.E.G. and Siemens, but it is understood that, as with other chlorinated compounds, conservatism and prejudice among customers have prevented its wider use.

No stabilisers are added to the material as their function of absorbing hydrochloric acid would be defeated by the formation of "chloride salts", which in turn become electrolysed. It was said to be very important to avoid contamination of Clophen T64 which may occur in transit and for this reason the use of leather for gaskets must be avoided.

4. Methods of use, precautions, etc.

No details were obtained on the method of impregnation used by I.G. for the construction of test-specimens of Clophen-impregnated capacitor. This was partly caused by the non-appearance, after the first day, of Engelhardt, but it is not considered likely that any novel processes are involved.

For information on the precautions necessary in the use of Clophen, see Section II, 5, above.

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12/19/47

From - B.I.O.S. Final Report No. 893
Item Nos. 1, 7, 22 and 31
Page xiv

Clophen is made in several grades, which correspond closely with their U.S. counterparts, A.50 being the grade most commonly used for capacitors.

Except for very rare use by Siemens and Halske for special orders, Clophen has not been used in Germany for small capacitors of the telecommunication type, but it has been used extensively by Siemens-Schuckert for power capacitors. It is interesting to note that the German forces used Clophen impregnated and filled tubular capacitors made by Ducati, of Milan, who obtained the impregnant from I.G. The prejudice against Clophen was, however, being gradually overcome and several manufacturers were contemplating an extended use of this material. Some of them considered it to be equal to or possibly better than mineral oil for operation at power frequencies.

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11/24/47

From - B.I.O.S. Final Report No. 893
Item Nos. 1, 7, 22, and 31
Pages 98, 99

(2) Clophen

The only chlorinated impregnant used by Siemens-Schuckert is Clophen, supplied by I.G. In power factor correction and high voltage applications it is considered practically ideal.

The only type of Clophen used in bulk is A.50, pentachlordiphenyl, (corresponding to Aroclor 1254). A.30 and A.40 have been tried and found unstable. A.60, A.70 and A.80 are too viscous to allow for good impregnation and have lower permittivities. Although the viscosity of A.50 at room temperature is much greater than that of oil, the viscosity at impregnation temperature is sufficiently low to give no trouble in attaining thorough impregnation.

A routine check on conductivity is made on each drum of Clophen, and formerly if the conductivity were too high, the Clophen was treated with powdered chalk to neutralise the free acid. As the result of continual pressure on I.G. over a period of years, the acid content had gradually diminished, and it is now possible without chalk treatment to work to the same rejection limit of resistivity as for oil, viz. 10^{13} ohm.cm. at 20°C. Drums of Clophen failing to pass the conductivity test are returned to I.G., unless urgently needed. A certain elasticity is permissible in view of the high standard set. Apart from conductivity, further acceptance checks are specific gravity (1.54-1.55) and permittivity (5.0-5.2). Permittivity is measured at 800 cycles and 20°C. on a Schering bridge at low voltage. The acceptance standards for Clophen A.50 are shown graphically in Appendix XXXIV.

Clophen costs about 1 M. 60 pfg./kg., and is therefore much dearer than oil (30 pfg./kg.). This extra expense is offset, however, by the use of a special design which reduces the amount of Clophen required. An advantage claimed for this impregnant is that is has a similar permittivity to paper (viz. 5).

2. Other impregnants studied.

As Clophen is considered nearly ideal, no serious attempts have been made to find superior materials or additives to improve its performance. This attitude is supported by service experience, in which no failures have been reported. It is realised, however, that on freezing discharges may occur in the contraction voids, which may result in decomposition of the Clophen and failure of the

dielectric. Tests were performed (see Section IV, 2, (3)) to check whether this degradation does in fact occur, and no failure was noted.

Nevertheless, I.G. supplied small samples resembling Clophen A.50 but having a lower melting point so that this possible source of failure could be investigated. Depression of the temperature at which the dipoles become immobile should raise the maximum usable frequency and hence broaden the range of applications by introducing those for audio-frequency furnaces.

Two of these I.G. samples, with references A.162M and A.162N, are, like Clophen, made from benzene, but have a lower chlorine content because of the presence of an additional alkyl group (C_nH_{2n+1}), and hence a lower density. Their advantage over Clophen A.50 is that the setting point (i.e. temperature of maximum dispersion and of capacitance diminution) is lowered from -8°C. to -17°C. The permittivity, viscosity and non-inflammability resemble those of Clophen A.50. The viscosity/temperature curves of these two samples, K.7 mineral oil and Clophen A.50., are compared in Appendix XXXV.

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11/19/47

III-B-2500

December 22, 1947

TO: PHOSPHATE DIVISION SALESMEN

SUBJ: Aroclor 5460 as Used in Ethylcellulose Lacquer

RE: Federation of Paint and Varnish Production Clubs Official Digest (October, 1947).

Aroclors used as plasticizers and resins impart excellent qualities to ethylcellulose.

Ethylcellulose is used in lacquers of various types, the ethylcellulose acting as a long chain polymer to impart toughness and flexibility and to increase the speed of drying.

Some of the principal uses for ethylcellulose lacquers are for the coating of high tension ignition cable; heat sealing lacquers for foil and paper where the slight discoloration which may develop in nitrocellulose lacquers on heat sealing is not permissible; linoleum lacquers where alkali resistance is essential; textile printing; printing inks, particularly in multicolor designs where hydrocarbon solvent does not soften or bleed into previously printed designs; aircraft lacquer for resistance to both heat and cold and to quick temperature changes; record lacquers, especially for home recorders; and in wood sealers where good sanding and flexibility are essential. A good pigmented ethylcellulose lacquer has been made, based on the following formula:

Ingredients	Parts (by weight)
Ethylcellulose N-22	6.4
Aroclor 5460	6.4
TiO_2	4.2
Menthylphenol	0.06
Toluene	67.0
Ethanol	16.0
	100.06

Benignus

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DSW 331687

III-G-300

From - Memo G.Y. Frankle/P.G. Benignus
March 11, 1948

Soil-Poison Concentrate

As described below, these materials are formulated into a water emulsifiable soil-poison concentrate carrying almost 80 per cent by weight of active ingredients.

Aroclor 1242	33.5%
Trichlorobenzene (Mixed isomers)	33.5
Pentachlorophenol	10.0
Isopropyl alcohol	3.3
Toluene or Xylene	16.7
Sterox SE*	1.5
Santomerse 3 Paste	1.5
	100.0%

* Other non-ionic type emulsifying agents such as Triton NE or Span and Tween can be used to replace the Sterox SE.

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DSW 331688

Chemical Abstracts

POSSIBLE USE OF AROCLORS

Vol. 41
Page 6867

U.S.P. 2,425,978 to Anderson and Coalahan, (Hercules Powder Company).

Foundry cores sprayed with 15% solution of chlorinated naphthalene in hydrocarbon solvent show greater scratch resistance than untreated cores. Cl_2 content remains from 40 - 80% Cl_2.

Particularly good for use with Al castings.

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12/2/47

From - B.I.O.S. Final Report No. 893
Item Nos. 1, 7, 22 and 31
Pages - 10 and 11

Much less trouble has been experienced with chlordiphenyl than with chlornaphthalene, probably because the former, being liquid, involves less physical contact in disposal.

(2) Prevention.

Very thorough washing of all areas of the skin likely to be contaminated with solid, liquid, or fumes has always been the basic principle in prevention of skin troubles, and is still looked upon as fundamental.

When fatty soaps ceased to be available, because of the diversion of fats to food, it became essential to insist upon the use of barrier creams, of which the best was one named "QUIMBO", made at Trommersdorf. This has now been replaced, for lack of supplies, by an alternative made by I.G., named "MITIGAL", which is considered inferior to Quimbo but satisfactory.

The Fissan firm put up a special powder named "SCHEFEL-PULVER" to act as a barrier, but this was found to be inefficient except to allay irritation on skin areas exposed to fumes only. For this purpose it still finds use at Leverkusen and at the Hydrawerke factory at Berlin.

A special point is made of fume extraction, and the minimum air speed away from the operator is 1 metre per second.

To check the efficiency of the precautions, a thorough examination is made every 4 weeks of every worker coming into contact with chlorinated hydrocarbons, and since the enforcement of the precautions, no deaths or symptoms of internal injury have occurred.

Since superfatted soaps have ceased to be available, a cleansing composition containing dichlor-methane has been devised. This is so powerful a detergent for chlorinated materials that barrier creams are considered unnecessary, but it is uncomfortably irritant to many skins.

(3) Cure.

When severe attacks of chloracne and internal sickness were encountered, in the early years, when the properties of chlorinated materials were not well understood, extensive rest periods had to be prescribed, together with a generous diet, entire absence of further contamination, and long periods in the open.

Since precautions have been taken to minimise the effects, only a few mild cases of chloracne have been met. These have been treated with a solution of acetic and salicylic acids in methylated spirits.

(4) Conclusions and Recommendations.

I.G. consider that neither toxicity nor skin affection need now be any deterrent to the use of chlorinated naphthalene, and that chlordiphenyl is if anything less troublesome.

Certain people, notably those with fair skins, show distinct allergy, and are best diverted to other work not involving contact.

Scrupulous cleanliness of the skin (including the face, neck, and arms) and of the clothing (especially undergarments) is essential and needs strict enforcement. Washing with hot water and good soap is sufficient, so long as the soap is superfatted, since the skin becomes sensitive to irritation by alkali.

Barrier cream should be used on those parts of the skin which come into contact with solid or liquid material, and the area of contact should be minimised by the use of protective clothing and gloves where possible. Areas of the skin which are contacted by fumes only, and particularly those chafed by clothing (e.g. the neck adjacent to the edge of the clothing) need protection by a suitable soothing powder.

Where fumes are generated, they should be drawn away from the operator at an air speed of at least 1 metre per second. It may be found essential to have two ducts, one at floor level and one overhead, with exhaust in two directions simultaneously.

In case the precautions are being evaded, or lest there should be some idiosyncrasy, monthly medical inspection is desirable.

rs
11/24/47

From - The Journal of Industrial
Hygiene and Toxicology
Volume 20, Number 2
February, 1938

MORPHOLOGICAL CHANGES IN THE LIVERS OF RATS RESULTING FROM EXPOSURE TO CERTAIN CHLORINATED HYDROCARBONS *

Chlorinated hydrocarbons, particularly chlorinated naphthalenes and chlorinated diphenyl, have been used extensively in certain industries. Their use in the manufacture and preparation of many types of electrical equipment is constantly increasing. Although it is known that some of these compounds cause acne, only recently has the possibility of more serious systemic effects been recognized.

The present paper describes the pathological changes observed in rats that had been exposed to various chlorinated naphthalene compounds and to chlorinated diphenyl.

Compound G (chlorinated diphenyl) was administered to two groups of animals in low concentrations. An average concentration of 0.57 mgms. per cu. m. was employed 16 hours daily for 134 days in the first experiment. In the second experiment (employing an average air concentration of 0.93 mgms. per cu. m.) the animals were exposed 8 hours daily for 143 days.

The present experiments demonstrate that chlorinated naphthalene compounds and chlorinated diphenyl are capable of producing marked liver damage in the white rat without demonstrable microscopic changes appearing in the other organs. Furthermore, the characteristics of the liver lesions resulting from comparable amounts of any given compound are the same, regardless of the method of administration (inhalation, feeding, or subcutaneous injection).

Of the various chlorinated hydrocarbons tested, chlorinated diphenyl gave evidence of being the most toxic. When administered by inhalation in very low concentrations (average 0.57 to 0.93 mgms. per cu. m.) liver cell changes were very pronounced after the first exposure period. The most striking change was the hyalinization of t the cell cytoplasm (see fig. 6, plate III). Such cellular alterations were essentially unchanged after a 2 month recovery period. In these animals small sublethal doses of carbon tetrachloride and alcohol uniformly produced extensive liver necrosis and was highly fatal to them (figs. 1 and 2, plate VII). Chlorinated diphenyl fed in small doses produced similar but more marked liver injury (see fig. 5, plate III). In large doses this compound was highly fatal. The liver changes in animals dying after short exposures were inconspicuous and consisted mainly of swelling of cells and active regeneration (see fig. 4, plate III). However, animals removed from exposure before being fatally

poisoned, subsequently developed hyaline degeneration of liver cells similar to that produced by prolonged administration of small doses of this compound.

Thus the results of the present study, as well as certain field studies that have been made (1) suggest that the solution of the industrial hazard involved is dependent largely on a reduction of the air concentration of these compounds to a level that will not produce liver damage. The present experiments indicate that lower air concentrations must be obtained in the case of the more highly chlorinated naphthalene compounds and chlorinated diphenyl than for trichlornaphthalenes if a safe environment for workmen is to be assured. Because of the pronounced toxic effect of small doses of carbon tetrachloride on the livers of animals already injured by exposure to chlorinated naphthalenes and chlorinated diphenyl, its use as a solvent for these compounds would appear to be very hazardous.

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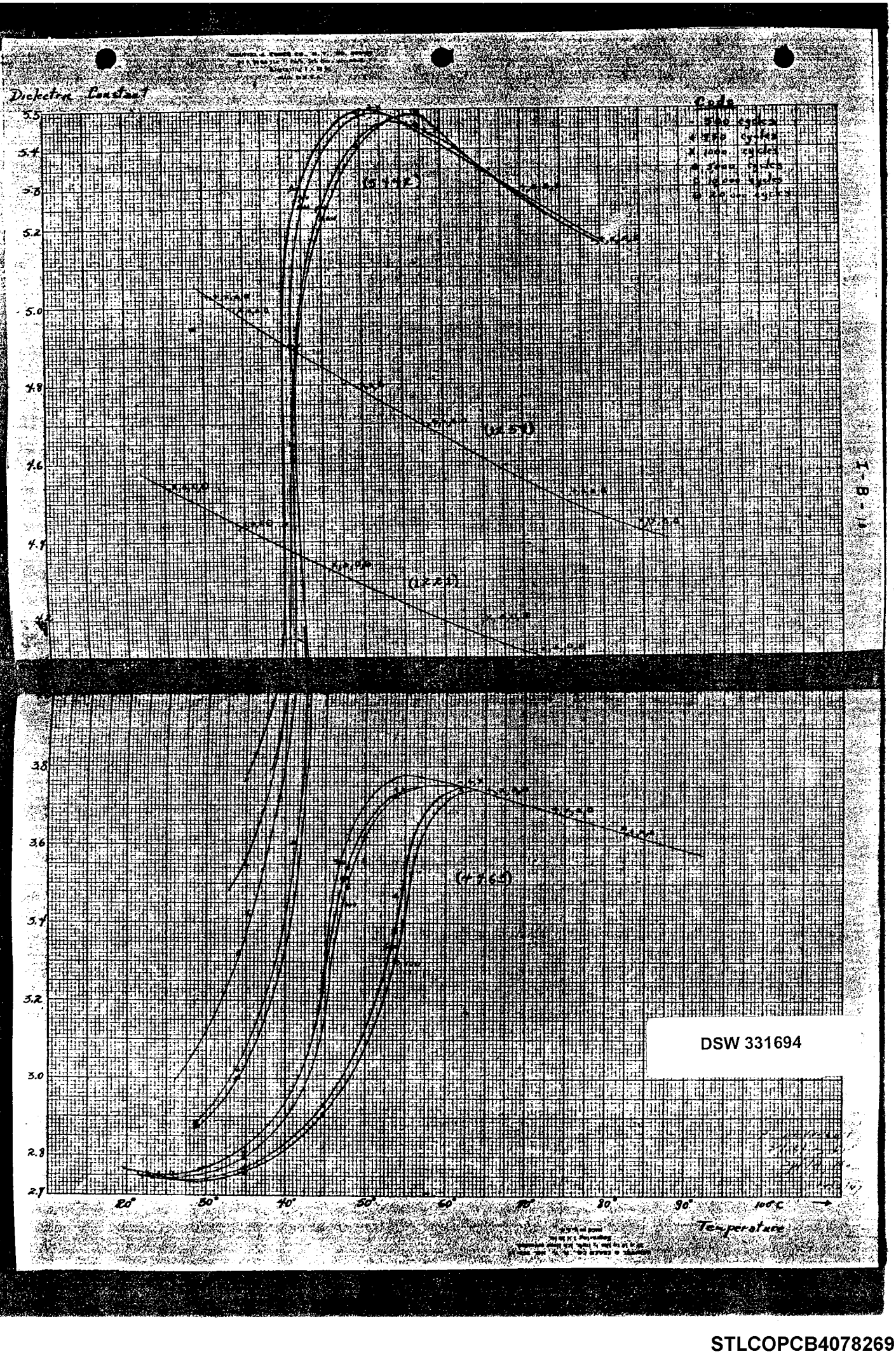

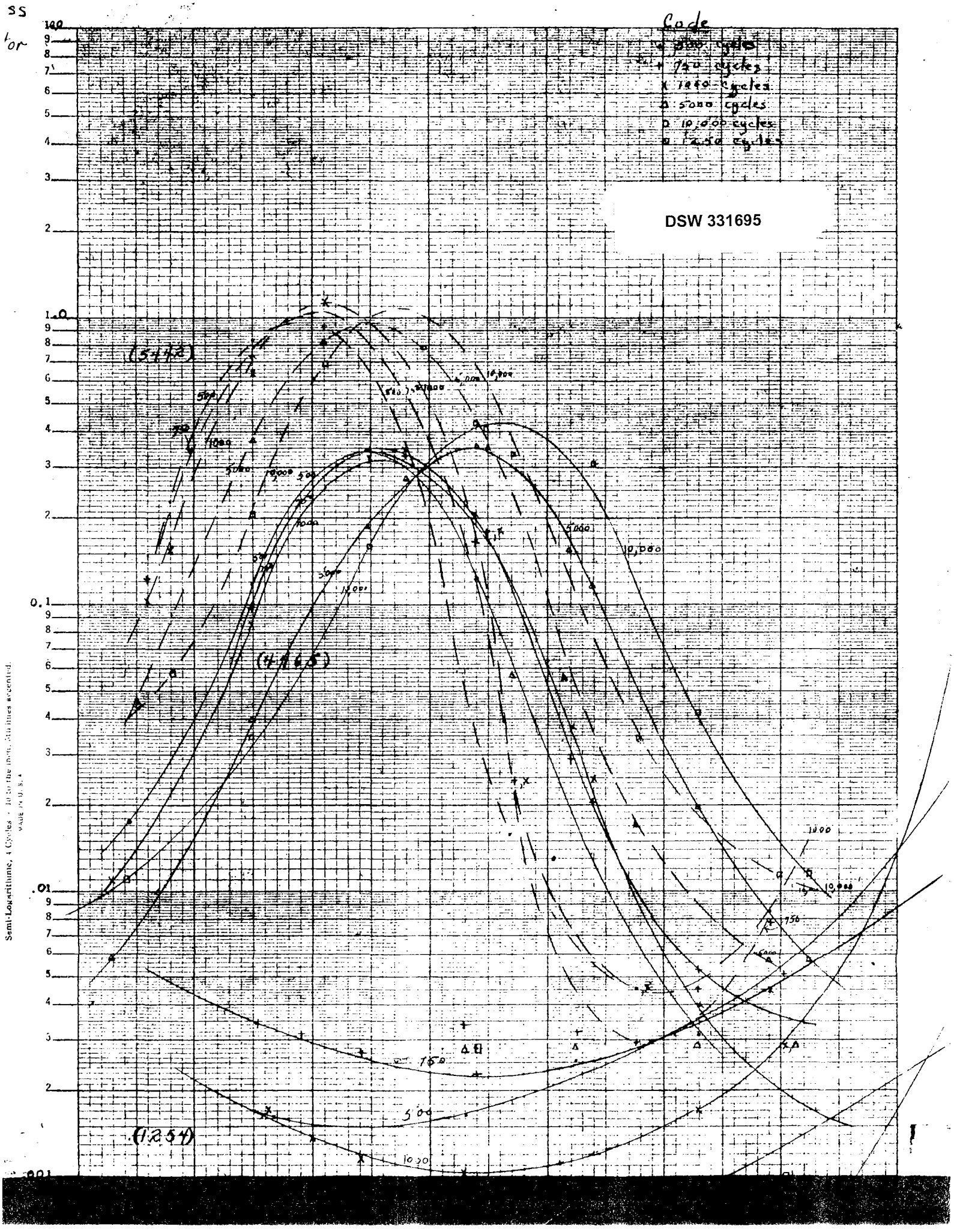

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