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

Eighteen copies were made of this report and distributed as follows:

No. 1. Research File
No. 2. R.L. Jenkins - C.B. Durgin
No. 3. R.R. Cole - R.S. Weatherly
No. 4. W.T. Durrett - F.P. LaBelle
No. 5. H.F. Weaver
No. 6. C.A. Hochwalt
No. 7. E.P. Rucker
No. 8. Edgar E. Hardy
No. 9. J.F. Reeves
No. 10. J.F. Reeves
No. 11. A.M. Ellenburg
No. 12. R.R. Knight
No. 13. Paul Logue
No. 14. P.G. Benignus
No. 15.
No. 16.
No. 17.
No. 18.

This is copy No. 15

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 331771

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 331772

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. 12-16-46; 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:

Copied by rs
4/8/48

DSW 331775

An 24

Page No. ____

STANDARD SPECIFICATION OF

MONSANTO CHEMICAL COMPANY
PHOSPHATE DIVISION
ANNISTON, ALABAMA

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

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

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

APPROVED BY (Initials)	R.L.J.:	A.M.E.:	J.F.R.:	F.A.B.
	12-17-46	12-18-46	12-19-46	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 SU3	

NOTES:

Copied by rs
4/8/48

DSW 331776

STLCOPCB4078351

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

Copied by rs
4/8/48

DSW 331777

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

Copied by rs
4/8/48

DSW 331778

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.

rs
5/11/48

DSW 331779

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.

rs
5/11/48

DSW 331780

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.	

Copied by rs
4/8/48

STANDARD SPECIFICATION OF

MONSANTO CHEMICAL COMPANY
PHOSPHATE DIVISION
ANNISTON, ALABAMA

PRODUCT: Arcolor 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.;	H.F.T.;	P.L.;	R.S.W.;	F.A.B.
	9-29-44	9-29-44	10-2-44	10-2-44	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:

Copied by rs
4/8/48

DSW 331782

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

Copied by rs
4/8/48

DSW 331783

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
Cleaning Distillation Cells	14-56-52

DSW 331784

Monsanto Chemical Company

Anniston Method No. 14-10-48

Specific Gravity of Aroclors

The temperature at which the gravities of liquid Aroclors are taken varies with the viscosity of the liquid. Note the temperature at which the gravity is to be taken for the Aroclor under test and heat the sample to 10°C. above that temperature. Pour the hot sample into the steam or hot water jacketed hydrometer jar provided for this test. Stir well with an accurate thermometer and adjust the temperature to the desired point by controlling the steam or hot water feed to the jacket. Continue stirring until the temperature remains constant for half a minute. Allow the hydrometer to sink into the liquid, then read the hydrometer scale at the point of the lower meniscus and record with the temperature.

Report the specific gravity to the nearest 0.001 unit.

DSW 331785

Monsanto Chemical Company

Anniston Method No. 14-13-48

SUBJECT: Total Chlorine in Aroclors

METHOD: Volhard Titration following Peroxide Fusion

Chlorine in Aroclors may be determined by fusion of the sample with sodium peroxide in a Burgess-Parr fusion cup, extracting the fusion with water, acidifying the water extract with nitric acid, and precipitating the chlorine by addition of an excess of silver nitrate. After filtration, the excess of silver nitrate is determined by titration against potassium thiocyanate, using ferric ammonium sulfate as indicator.

Apparatus

The fusion cup used is the Burgess-Parr Sulfur Bomb No. 3 for flame ignition. A lead gasket is used. For ignition, the bomb is suspended through a 1-3/16" round hole in a 1/8" transite plate. This allows the fusion cup to extend through the plate for about 5/8 inch. Ignition is effected by strongly heating the bottom of the fusion cup with the full flame of a Meker burner for two minutes.

The bomb, gaskets, and the sodium peroxide are obtained from the Parr Instrument Company, Moline, Illinois.

Charge for Fusion

The fusion mixture is made up of about 15 grams (one metal scoop) of sodium peroxide and 0.3 grams of finely powdered cane sugar. The reagents should be free from chlorine, or a blank run and corrected accordingly. The fusion mixture is well mixed by placing the ingredients in a small glass stoppered bottle and shaking vigorously. A measuring spoon for the sugar is convenient.

SOLID AROCLORS: The non-crystalline type are weighed in the form of small pellets. These are prepared by heating the Aroclor until a consistency is reached as will permit dropping it from a glass stirring rod onto a tinned surface (a can top), each drop forming a pellet. When cool these pellets can be removed from the surface by inserting a spatula under them. .4 grams are used. The pellets are brushed into the bottom of the fusion cup and the fusion mixture placed on top of them. After tightening the lid, the charge is ready for fusion.

The crystalline type Aroclors are weighed in the powdered form. .4 grams being used. They are charged in the same manner as the non-crystalline type.

DSW 331786

LIQUID AROCLORS: .20 to .30 grams are weighed by dropping from a stirring rod onto a piece of thin hemispherically shaped glass, or 1/2 gelatin capsul (Gelatin Capsules No. 00, United Drug Company, Boston - St. Louis), which has been just previously tared, and which remains on the balance. It is necessary to heat the more viscous Aroclors to "dropping" consistency. The piece of glass then easily slides off the balance pan into the fusion cup. Cover with the fusion mixture.

The following quantities of sample, sugar, and $AgNO_3$ are used for the respective chlorine contents:

Cl Content	Weight of Sample	Amount Sugar	$AgNO_3$
0% - 30%	0.3 grams	0.3 g.	50 ml.
30% - 45%	0.3 grams	0.3 g.	50 ml.
45% - 58%	0.3 grams	0.3 g.	50 ml.
58% - 66%	0.3 grams	0.3 g.	75 ml.
66% - 70%	0.3 grams	0.3 g.	100 ml.

Procedure

Place the fusion cup, which contains the prepared sample and fusion mixture, in the transite ignition plate and apply the full flame of the Meker burner to the bottom of the fusion cup for 2 minutes.

(CAUTION: Do not stand too near the fusion during ignition. The use of a safety shield is recommended.)

Then remove the flame and cool under the tap. When cool remove screw cap. Thoroughly rinse the cup cover with water, collecting the rinsings in a clean 400 ml. beaker.

Then place the fusion cup on its side in the beaker and cover with a watch glass. 50 - 75 ml. will be in the beaker from rinsing the cap, and this is sufficient to decompose the charge. Remove the cup and rinse well. When decomposition is complete, rinse off cover-glass and add pure HNO_3, with stirring, until acid is present in excess to the extent of about 10 ml. (About 50 ml. of acid are required.)

Add exactly 50, 75, or 100 ml. of standard silver nitrate solution from a pipet (depending upon chlorine present) and stir to effect coagulation of silver nitrate precipitate. Filter with suction through an asbestos pad on a 1-1/2" perforated porcelain plate and wash beaker and filter 4 times with small portions of cold water. Allow the filter to drain completely between washings. Transfer the filtrate back into the 400 ml. beaker and rinse the flask twice, adding the rinsings to the beaker.

Add 5 ml. of ferric iron indicator to the solution and titrate with standard KCNS solution until a distinct pink tint is just obtained.

DSW 331787

Calculation of Chlorine Content

Subtract the volume of KCNS required from the volume of KCNS required to titrate the amount of standard $AgNO_3$ solution used. This will give the volume of KCNS equivalent to the chlorine in the sample. Multiply the volume so obtained by the chlorine value of each ml. of KCNS and divide by the weight of sample taken.

For example: If 50 ml. of silver nitrate solution is equivalent to 61.4 ml. of KCNS solution and the value of 1 ml. of KCNS solution is .003770 gram Cl; then if it is found that 0.3 gram of sample shows a KCNS titration of 10.4 ml. the percent of chlorine is:

$$\frac{(61.4 - 10.4) \times .003770 \times 100}{.3} = 64.09\% \text{ Cl}$$

In case the fusion mixture or other reagents contain chlorine, the amount must be determined and deducted from the chlorine found.

Solutions Required

Standard $AgNO_3$ Solution: Dissolve 22 grams of silver nitrate in each liter of water. Protect the solution from light.

Standard KCNS Solution: Dissolve 10 grams of KCNS in 1 liter of water.

Ferric Iron Indicator: Use a saturated solution of ferric ammonium alum, about 50 g. per 100 ml. of water.

Pure Nitric Acid: Stock acid suffices provided it is colorless. It can be boiled in a beaker until colorless if necessary using 600 ml. HNO_3 and 300 ml. of H_2O.

Standardization of Solutions

Weigh 0.3 and 0.15 gram portions of pure dry NaCl into separate 600 ml. beakers. Add 250 ml. of distilled water to each and 10 ml. of pure 50% nitric acid. When solution is complete, add 50 ml. of standard $AgNO_3$ solution from pipet. Stir well and filter through an asbestos mat on a perforated porcelain plate, using suction. Wash filter and transfer the filtrate back to the beaker in the same manner as when working with a sample. Titrate the filtrates with standard KCNS solution, using ferric iron indicator.

The value of 50 ml. of $AgNO_3$ solution in terms of KCNS solution is found by subtracting the ml. of KCNS solution in titrating the 0.3 gram of NaCl from twice the number of ml. of KCNS solution used in titrating the 0.15 gram of NaCl.

This value is checked by titrating 50 ml. of $AgNO_3$ solution added to 400 ml. of water and 10 ml. of pure 50% HNO_3 with KCNS solution. This titration should agree very closely (± 0.1 ml.) with the value found from the titration of NaCl. The titration must be made slowly to avoid drainage error.

Since pure NaCl contains 60.66% Cl by theory, the value of the KCNS in terms of chlorine may be found by dividing the weight of chlorine in the NaCl taken by the ml. of KCNS equivalent to the silver nitrate required.

Example:

> Two 0.3 gram portions of NaCl show KCNS titrations of 13.11 and 13.13 ml.
>
> Two 0.15 gram portions of NaCl show KCNS titrations of 37.28 and 37.24 ml.
>
> The KCNS equivalent to 50 ml. of $AgNO_3$ is then (37.28 + 37.24) - 13.12 or 61.4 ml.

By titration of 50 ml. of $AgNO_3$ against KCNS solution, it is found that 61.36 ml. are required. This agrees within .04 ml. of the value obtained from the NaCl titrations.

The chlorine value of the KCNS is then, .6066 X .3 divided by 61.4 - 13.12 or .003770 grams Cl per ml. of KCNS.

The chlorine value may also be calculated from the titration of 0.15 gram of NaCl. 0.6066 X .15 divided by 61.4 - 37.26 gives .003770 grams Cl, per ml. of KCNS.

Precautions

1. The fusion materials have explosive properties if handled improperly; consequently care must be taken to use safe proportions, to secure a good mixture free of large lumps, and to properly seat the cover of the fusion cup. The fusion mixture must be kept away from water or moist air, either of which may ignite the charge. The fusion mixture should not be ground to reduce lumps.

2. The method as described is not applicable to volatile organic compounds unless precautions are taken to avoid loss of sample during weighing.

Effect of Variables

1. On account of the high chlorine content of many of the Aroclors and the small amount of sample taken, great accuracy is required in weighing, transfer, and mixing of the sample. The pipet and buret for measuring the standard solutions must be very clean to avoid drainage errors.

2. Fusions which show black carbon deposites on the cover and side of the fusion cup may or may not give the full chlorine content. If a carbon deposit is found, a second fusion should be made using a smaller sample or reducing the amount of sugar in the charge or both.

3. The temperature at which the standard solutions are standardized should be noted. In case room temperatures vary from this temperature, appropriate volume corrections should be made.

4. The fusion mixture materials should be essentially free of chlorine. The chlorine content may be determined by making a blank fusion, that is, without addition of sample, and titrating in the usual way. Five ml. of $AgNO_3$ may be added instead of 50 ml. portions of $AgNO_3$ solution in like volumes of solution and nitric acid.

References

Beamish: Determination of Organic Halogens, Ind. Eng. Chem., Anal. Ed., 6 352 (1934).

FAB:cm
1-29-45

Copied by rs
4/20/48

DSW 331790

Monsanto Chemical Company

Anniston Method No. 14-17-48 ST

SUBJECT: Softening Point of Solid Aroclors

METHOD: Ball and Ring

ST Ref. ASTM - 1951 Supplement, Part 3, page 225

This method is a modification of the A.S.T.M. standard method of test for softening point of bituminous materials, serial designation: E 28-~~38T~~. ~~The method differs from the standard method in that the~~ rings are larger than specified. A two ring support is also used in order that two tests may be made simultaneously.

Apparatus

The apparatus consists of the following: (a) Two tapered brass rings, 5/8" inside dia. at bottom, ~~11/16"~~ inside diameter at top and 1/4" deep: thickness of wall 3/32" (b) Two steel balls, 3/8" diameter weighing 3.45 to 3.55 grams each. (c) A 800 ml. Griffin low form beaker. (d) A ring support for the two rings having a brass plate exactly one inch below the plate supporting the ring. (e) ~~A 300°C. A.S.T.M. low distillation thermometer graduated 1°C.~~ a ASTM Low or High Softening Point thermometer.

Preparation of the Sample

The sample shall be melted and stirred thoroughly, avoiding overheating or incorporating air bubbles in the mass and then poured into the ring so as to leave a slight excess on cooling. Since the Aroclors shrink considerably on cooling, the ring should be well filled, nearly to overflowing. A little of the excess should be drawn over the top of the ring at several points so as to prevent the cooled Aroclor from dropping out of the ring. In the same way, the second ring is filled with a standard Aroclor of known softening point. The standard Aroclor should have a softening within at least 10 to 15° of the Aroclor being tested.

The rings while being filled should rest on a clean can lid or on a brass plate which has been amalgamated to prevent the Aroclor from adhering to it. The Aroclor in the rings should be fully cooled and hardened before proceding with the test.

Procedure

Add cool solution (employ water for softening point between 0-80°C, glycerin for between 80-200°C., mineral oil for above 200°C.) to the beaker until the surface of the solution is 2" above the plate holding the rings when the ring support is suspended in the beaker. Place the rings containing the Aroclor to be tested and the standard Aroclor on the ring support. Place a ball on the center of the upper surface of the Aroclor in each ring. Suspend the thermometer so that the bottom of the bulb is level with the bottom of the rings and just midway between the two rings. The thermometer is held in position by a one holed stopper mounted into the center hole of the assembly, Fig 1-D

DSW 331791

Place beaker and apparatus on a ~~6 inch round hot plate~~ wire gauze sheet supported by a tripod and heat with a gas burner at such a rate that the temperature is raised 5°C. each minute. [illegible] The temperature recorded by the thermometer at the instant the Aroclor touches the bottom plate is reported as the softening point. The heating is continued until both Aroclors have dropped to the bottom plate. No corrections are made for emergent stem.

Notes

1. The standard Aroclor is run along with the sample under test in order to compensate for variations in rate of heating, dilution of glycerin, and thermometer variations.

2. The softening point of the standard sample is determined by making several determinations using fresh glycerin and carefully checking the rise in temperature of the glycerin so that it is as near as possible to 5°C. per minute.

3. The glycerin may be used repeatedly for the tests after removal of the Aroclor.

4. Benzol is used for cleaning the rings and balls.

5. Water can be used instead of glycerin for softening points up to 90°C.

6. For softening points above ~~100°C~~ 90°C, well boiled glycerin should be used. The usual glycerin is not satisfactory above 125 to 130°C, it boils with loss of water while the temperature remains practically constant. It is well to have a supply of high-boiling glycerin on hand to be used only for softening points above ~~100°C~~. 90°C

7. Air bubbles on the rings and balls during the test should be avoided as far as possible.

8. The rate of rise of temperature of the glycerin shall be uniform for each minute after the first 3 minutes of heating and not averaged over the period of the test.

FAB;cm
1-31-45

Copied by rs
4/20/48

JWC
6/54

DSW 331792

Monsanto Chemical Company

Anniston Method No. 14-21-45

SUBJECT: Determination of Iron in Aroclors

Iron in Aroclors may be determined by extracting a benzol solution of the Aroclor with dilute hydrochloric acid until further extractions show no appreciable iron content. The iron in the combined extracts is then determined by dichromate titration.

Procedure

Transfer 5 - 10 grams of sample to a clean separatory funnel of about 150 ml. capacity. Add about 50 ml. of benzol and shake until the sample is in solution.

When solution is complete, add 10 ml. of 1:1 iron-free HCl and shake for about 2 minutes. Let stand until separation of the two layers is complete, then draw off and preserve the acid extract. Add another 10 ml. of 1:1 HCl and shake again. Draw off the acid layer and combine with the first extract. Continue until HCl layer is clear. Determine the iron by usual dichromate titration method.

FAB:cm
1-31-45

Copied by rs
4/21/48

Monsanto Chemical Company

Anniston Method No. 14-24-48

SUBJECT: Flash and Flame Points

METHOD: Cleveland Open Cup

The flash and flame points of Aroclor shall be determined in the Cleveland Open Cup Tester, following the procedure described in ASTM D 92-33.

Apparatus

The cup shall be supported by a metal plate 1/4" (.635 cm) in thickness and 6 inches (15.24 cm) in width. The plate shall be of brass, cast iron, wrought iron, or steel. In the center of the plate there shall be a plane depression 1/32" (.079 cm) in depth, and of just sufficient diameter to fit cup. There shall be a circular opening 2 3/16" (5.50 cm) in diameter, cut thru the plate, centering with the center of the above mentioned depression. The plate shall be covered with a sheet of hard asbestos board 1/4" in thickness, and of the same shape as the metal plate. There shall be cut in the center of the asbestos board a circular hole just fitting the cup. Heat may be supplied from any convenient source. The use of a gas burner, electric heater, or alcohol lamp is permitted, but under no circumstances are products of combustion or free flame allowed to come up around the cup. The source of heat shall be centered under the opening in the plate and shall be of a type that will not produce local superheating. If a flame heater is used, it may be protected from drafts or excessive radiation by any suitable type of shield, that does not project above the level of the upper surface of the asbestos board. The thermometer shall conform to the requirements of thermometer No. 8. (See ASTM table of thermometers).

Procedure

The thermometer shall be suspended or held in a vertical position by any suitable device, the bottom of the bulb shall be 1/4 in. (.635 cm) from the bottom of the cup, and above a point half way between the center and back of the cup. The cup shall be filled with oil to be tested in such a manner that the top of the meniscus is exactly at the filling line at room temperature. The surface of the oil shall be free from bubbles. There shall be no oil above the filling line or outside of apparatus. The test flame shall be approximately 5/32" (.397 cm) in diameter.

The test flame shall be applied as the temperature read on the thermometer reaches each successive 5°F mark. The flame shall pass in a straight line, (or on the circumference of a circle having a radius of at least 6 inches.) across the center of the cup and at right angles to the diameter passing thru the thermometer. The test flame shall, while passing across the surface of the oil, be in the plane of the upper edge of the cup. The time for the passage of the test flame across the cup shall be approximately 1 second.

DSW 331794

The oil shall be heated at a rate not exceeding 30°F per minute temperature rise till a point is reached approximately 100°F below the probable flash point of the oil. Thereafter the rate of heating shall be decreased and for at least the last 50°F before the flash point is reached the rate shall be not less than 9°F. or more than 11°F per minute.

The flash point shall be taken as the temperature read on the thermometer when a flash appears at any point on the surface of the oil. The true flash must not be confused with a bluish halo which sometimes surrounds the test flame.

After determining the flash point, the heating shall be continued at the specified rate of 9°F to 11°F per minute, and application of the test flame shall be made at the specified intervals until the oil ignites and continues to burn for a period of at least five seconds. The method of application of the flame shall be the same as for flash point. The temperature read at the time of the flame application, which causes burning for a period of five seconds or more, shall be recorded as the flame point.

The flash point and flame point tests shall be made in a room or compartment free from air drafts. The operator shall avoid breathing over the surface of the oil. It is desirable that the room or compartment may be darkened sufficiently so that the flash may be readily discernible.

Note

Aroclors 1248, 1254, 1260, and higher chlorinated Aroclors do not have a distinct flash or flame point below their boiling temperatures.

FAB:cm
1-26-45

Copied by rs
4/21/48

MONSANTO CHEMICAL COMPANY
Anniston, Alabama

Analytical Laboratory

Anniston Method Number 14-4-52

Test Method: Viscosity – Saybolt Universal

Reference: Method No. 11,433-52 WGK GM 25

1. Take a scrupulously clean, dry 60 ml. viscosity receiving flask from the oven and allow it to cool to room temperature while the viscosimeter is being cleaned. (Use only receiving flasks which have been standardized and found to be within ± 0.05 ml. of 60.00 ml.)

2. Clean the viscosimeter tube as follows:

   A. Insert the cork, which should be clean and in good condition, in place in the bottom of the tube.

   B. Carefully pour enough benzene into the tube so that the latter is filled and the liquid overflows into the gallery. Do not allow any benzene to spill down into the oil bath, as the vapors will affect later viscosities. Caution: While using benzene for cleaning the viscosimeter, be sure all circuits of the instrument are turned off.

   C. Using a thermometer fitted with a holder to prevent it touching the bottom of the tube, stir the mixture well so that any adhering material is dissolved. Be sure the entire inner surface of the tube and gallery is wet. Pull the cork to drain the tube. With a clean withdrawal tube draw the excess liquid from the gallery and discharge it directly into a beaker – not into the viscosity tube.

   D. Repeat steps A. – C. inclusive.

   E. Permit the viscosimeter tube to drain and dry. ABSOLUTELY DO NOT INTRODUCE A CLOTH OR KLEENEX INTO THE TUBE OR GALLERY FOR BLOTTING UP RESIDUAL LIQUID.

   F. Flush ca. 80 ml. of the well-shaken (and heated if needed to insure fluidity) sample to be tested through the apparatus and follow step C. above.

   G. Drain this out, and again flush with a fresh portion of sample to make certain any last drops of previous material have been rinsed out.

   H. Shine a pen-light up through the orifice while looking down into the tube. The orifice should be free of fibers or any other obstruction. If these are present, they should be removed by flushing with sample only, not by use of wires. If flushing with sample fails, inform the supervisor.

DSW 331796

ADDITIONAL PRECAUTIONS:

A. See that the bath oil, when hot, is not less than 1/4" above the level of the overflow rim of the gallery of the tube.

B. The bath oil should be a grade of light-colored mineral oil which has a viscosity of 40 ± 5 Saybolt seconds at 210°F and should be replaced when it displays a definite darkened color.

C. For viscosities to be run at 100°F, the bath oil temperature shall not exceed 100.25°F (± 0.05°F for 10 Min.). For viscositites determined at 130°F, the bath temperature shall not exceed 130.50°F (± 0.05°F for 10 Min.). For viscosities determined at 210°F, the bath shall not exceed 212.0°F (± 0.10°F for 10 Min.).

D. Use only thermometers standardized to the nearest 0.01°F against a National Bureau of Standards thermometer.

E. Use only the stopwatch provided for timing purposes, which should be accurate to within 0.1 per cent when tested over a 60 minute period. Electrical timers must not be used unless the available power source is known to be of sufficiently accurate frequency. The stopwatch must be left in the holder provided, since variations in its position can cause error.

F. Never use the plunger, commonly provided, for cleaning the instument and never expose the viacosimeter to a draft during a determination.

3. Blot the cork dry with a lint-free cloth and insert it in the tube. Pour ca. 150 ml. of sample into a scrupulously clean beaker which has been rinsed with sample, and heat the contents to not over 7°F hotter than the temperature of test. Do not use a sample which has overheated, even if it is then cooled to within the prescribed range.

4. Pour enough heated sample into the tube that it ceases to overflow into gallery. All samples must be strained through the 100 mesh screen which has been carefully flushed with sample.

5. Stir the sample with the standardized thermometer (with attached holder) until its temperature has remained constant within ± 0.02°F of the desired temperature for one full minute (with constant stirring).

NOTE: Stirring is a very critical point, especially in the case of more viscous liquids such as Aroclor 1260. Use the exact technique described as follows:

Stir the sample with the thermometer, continuously in the same direction, at a measured rate of three revolutions per second. Alternately sweep the thermometer against the walls of the tube for five revolutions and then stir in the center of the tube for three revolutions. Do not deviate from the prescribed rate of stirring or the practice of stirring five revolutions at the walls of the tube, three revolutions at the center, five at the walls, three at the center and so on for the one-minute period specified.

Regulate the temperature of the sample while stirring, by adjusting the oil bath until the desired reading holds constant within ± 0.02°F throughout the required interval. Do not stir by moving the thermometer up and down unless you desire to cool the sample, and under no circumstances stir in this manner during the one-minute timed interval.

6. Remove the surplus sample from the gallery, with the scrupulously clean withdrawal tube which is inserted at one point in the gallery without touching the over-flow rim, and which removes sufficient sample that the level of sample in the gallery is below the level of sample in the oil tube proper. Do not rotate the withdrawal tube around the gallery.

7. Place the receiving flask in position so that the stream of oil from the outlet tube strikes the neck of the flask.

8. Snap the cork from its position, and at the same instant start the stopwatch.

9. Stop the stopwatch at the same instant that the bottom of the meniscus of the sample reaches the mark on the neck of the receiving flask.

10. Examine the end of the cork to see if it has become moistened with sample. This would denote an incomplete seal and a premature seepage of sample through the orifice which would produce erroneous results.

The time in seconds, after applying the proper calibration correction of the tube is the Saybolt Universal Viscosity.

Different operators in different laboratories should agree within 0.5% on all readings.

Report the results to the nearest 0.1 seconds.

Reference: ASTM D-88-44

OPERATION OF THE TAG VISCOSIMETER

The viscosimeter is heating when the lamp is off. Do not go away and leave the "quick heat" on. The "Quick heat" should be turned off when the temperature of the bath is within 1 degree of the desired bath temperature. The viscosimeter should be allowed

30 minutes to come to temperature equilibrium before making a test. This will allow the metal parts of the viscosimeter to come to temperature equilibrium with the bath. Note: Every now and then, trouble will be found in getting the bath to hold the proper temperature.

Monsanto Chemical Company

Anniston Method No. 14-31-48

SUBJECT: Distillation Range of Liquid Aroclors

METHOD: A.S.T.M. D-20 with Modifications

The apparatus, consisting of flask, condenser tube, shield, and thermometer, is exactly the same as described under A.S.T.M. test D-20 "Distillation of Bituminous Materials suitable for Road Treatment". EXCEPT

Attach a 6 inch auxiliary thermometer (0 - 150°C.) to distillation thermometer. Place bulb half way between top of cork and probable average of distillation range. Cover both thermometers with a 3/4" diameter glass tube to insure a uniform temperature correction for the exposed stem, barometric pressure and thermometer error.

The procedure is changed only to the extent that thermometer readings are taken when specified percentages (usually 10, 50, and 90%) of Aroclor have been distilled instead of following the A.S.T.M. procedure of weighing the distillate between specified thermometer readings.

In testing liquid Aroclors, 100 gms. of sample are weighed into the distilling flask and distillate received in a tared flask or beaker resting on pan of a Torsion balance with 100 gram scale.

Procedure

Weigh out 100 grams of sample into the distilling flask. Assemble apparatus as described under A.S.T.M. D-20. Insert thermometer (A.S.T.M. high distilling 0 - 400°C.) thrd cork in the neck of the flask so that the top of the bulb is level with the lowest point of juncture of the tubulature and neck of the flask. Apply heat to the flask supported on two sheets of 20 mesh wire gauze so that the first drop comes over in from 5 to 15 minutes.

Conduct distillation at rate of 50 to 70 drops per minute. Collect distillate in a 250 ml. beaker tared on a balance. Take temperature readings at 1%, 3%, 5%, 10%, 20%, 30%, 40%, 50%, 60%, 70%, 80%, 90%, 93%, 95%, 96%, 97%, and dry.

Corrections for emergent stem and pressure are applied to the thermometer readings if required. Take correction readings at first drop, 10%, 50%, 90%, and dry. Report the temperatures to the nearest 1°C.

Example: (Exposed stem in degrees) X (temperature difference in degrees) X 0.000158 = degrees stem correction = T_1. The exposed stem is read from the top of the cork. The temperature difference is the reading of the thermometer minus the temperature of the auxiliary thermometer.

DSW 331800

0.00012 $T_b \Delta p$ = barometric correction = T_2 T_b = normal boiling point in degrees absolute; Δp = change in pressure from 760 mm. Hg.

The correction is added if barometer is below normal and subtracted if the barometer is above normal. (MacDougall - "Thermodynamics and Chemistry" pp. 113). Corrected Temperature = Observed temperature + $T_1 \pm T_2$.

FAB:cm
1-29-45

Copied by rs
4/22/48

DSW 331801

Monsanto Chemical Company

Anniston Method No. 14-32-48

SUBJECT: Evaporation Test of Liquid Aroclors

METHOD: 6 Hours Heating at 100°C. ASTM D6-39T - Modified

Procedure

Weigh on a rough balance about 50 grams (± .5 gm.) of the well mixed Aroclor into an accurately tared tin box, 55 mm. dia. X 35 mm. deep (3 oz. Gill-style ointment box, deep pattern), Fisher #1-820. Let stand until box and sample are at room temperature, then weigh accurately.

Place the box in a ventilated convection oven maintained at 100°C. ± 1° for 6 hours. Remove, cool in desiccator to room temperature and accurately reweigh. From the loss in weight calculate the evaporation in per cent. Report to second decimal place only.

Notes

1. Be sure box is at room temperature whenever exact weight is taken.
2. The oven should not contain other samples which might interfere with evaporation.
3. Because the evaporation loss is sensitive to temperature, the air bath must be closely maintained at 100°C.

FAB:cm
1-29-45

Copied by rs
4/23/48

DSW 331802

Monsanto Chemical Company

Anniston Method No. 14-34-48

SUBJECT: Refractive Index of Liquid Aroclors

METHOD: Abbe Refractometer

Refractive Index

The refractive index of any medium is defined as the ratio of the velocity of light in air to the velocity of light in that medium. It is measured by the ratio of the sine of the incident angle to the sine of the angle of refraction. The denser the medium, the greater is the refraction toward the normal (higher refractive index). Because the refractive index is characteristic of each substance, it is useful in identifying liquids and verifying their purity, in determining the molecular structure of organic compounds, and in some quantitative analyses of mixtures. As applied to Aroclor testing refractive index provides a verification of composition and purity.

Apparatus

Abbe Refractometer with accessories, Bausch and Lomb, Cat. #2550, Serial No. 377. Calibrated to read directly in terms of refractive index of the D line (sodium) at a temperature of 20°C.

Procedure

Operators should be familiar with the Bausch and Lomb "Directions for Use" and practice the manipulation as there described before attempting determinations on Aroclor. The abridged directions below provide a working outline but at the expense of omission of essential explanatory details which are contained in the B. and L. directions.

Screw thermometer into its socket in water jacket of upper prism. Open prisms and check their condition. Clean if they are soiled, streaked, or spotted. With lower fixed prism horizontal, place on it 2 or 3 drops of the Aroclor from a stirring rod - sufficient to fill the spaces between prisms when clamped together. Close prisms and lock with lock nut.

Adjust the instrument and mirror to reflect white light into the refractometer. No spots on air pockets should be visible. Connect water jacket to source of constant temperature water, usually the tap, and pass water until the thermometer has been constant to 0.2°C. for at least 5 minutes at 25°C. The Aroclors flow more easily at 25°C., hence the choice of this temperature.

With prisms illuminated, rotate the prisms by means of the index arms until the border of the light and dark fields passes exactly thru the intersection of the cross hairs. If the border line is fringed with color, rotate the compensator until the color disappears. If the boun-

dary is not sharp, adjust mirror until a distinct half shadow is obtained. If the line or the cross hairs are blurred, adjust eyepiece until a good focus is obtained. Secure final fine adjustment by means of the slow motion screw.

Read the refractive index, estimating to the fourth decimal place. If the temperature of the water jacket is other than 25°C., correct to 25° by use of the correction factor, + 0.00044 per °C. This factor is applicable only to Aroclors 1248, 1254, 1260, and 1262. It was found by careful measurement on each Aroclor thru the range 10° to 50°C. The correction is subtracted when the working temperature is below 25°C. and added when above 25°C.

Example: If the refractive index is found to be 1.6422 at 15°C., the index corrected to 25°C. is 1.6422 - (10 X .00044) = 1.6378.

Care and Calibration of Instrument

Immediately after use, clean the prisms with swabs of pure cotton dipped in benzol, finally wiping dry with a soft cloth. The prisms, especially the upper one, tarnish and scratch easily. Consequently great care must be taken in cleaning. Before use the prisms should always be inspected and recleaned if necessary. When not in use, the refractometer should be kept in its closed case.

For practice or for checking performance, distilled water is useful. Water has a refractive index of 1.3330 at 20°C., with temperature coefficient of 0.0001. A test piece of special glass of known refractive index is supplied with the instrument for calibration and adjustment if necessary of the position of the index arm.

Refractive Index of Aroclors - Typical

Careful measurements at 25°C. have given the following typical values for Aroclors.

Aroclor 1248	1.6295
Aroclor 1254	1.6376
Aroclor 1260	1.6460
Aroclor 1262	1.6482

Substantial departures (over 0.0010) from these values should be investigated immediately as they - if correct - indicate substantial error in the composition of the Aroclor or contamination with other materials.

FAB:cm
1-27-45

Copied by rs
4/23/48

DSW 331804

Monsanto Chemical Company

Anniston Method No. 14-35-48

SUBJECT: Resistivity of Liquid Aroclors

METHOD: Resistance Measurements between Conductors

Apparatus

Megohm Bridge: General Radio Company Type 544B, AC operated - 500 volts, serial No. 171. This is a direct-current Wheatstone bridge for measuring high resistances, bridge balance being obtained thru use of a vacuum tube voltmeter. Range: 0.1 megohm to 10,000 megohms, covered by a dial and 5-position multiplier switch. A resistance of 1,000,000 megohms can be detected.

Test Electrodes: Two concentric nickel cylinders (or brass, nickel plated) with feet. Obtained from General Electric Company. The inner electrode has outside diameter of 2.8" and height of 3.25", with area of 184 sq. cm. The outer electrode has inside diameter of 3" and like height of 3.25", with area of 198 sq. cm. The distance between electrodes is therefore 0.1 inch or 0.254 cm. By theory the electrode constant, area/length, is 191/0.254 or 752 where 191 is the average area. In practice the cell constant supplied by General Electric Company is used. For the electrodes now in use, the cell constant was given as 815.

Glass Plate: Pyrex, about 3-1/2" diameter with concentric grooves to assist in spacing the electrodes. Obtained from General Electric Company.

Oven: Cenco-DeKhotensky drying oven with two holes in back wall for porcelain tubes for lead wires. Holes must avoid oven heating elements which occupy 2" paths vertically and horizontally intersecting at the center of the circular wall.

Lead Wires: Provide 300 ohm Amphenol twin conductor cable to connect the electrodes in oven to the posts of the bridge. The portions exposed to oven temperature are bared and then covered with porcelain beads. Provide also a flexible lead to ground the bridge to a water pipe. Clean the leads, beads, and porcelain tubes periodically.

Assembly of Test Cell

The grooved glass plate is placed in an 800 cc. beaker. The clean electrodes are placed in the grooves of the glass plate with the connector posts directly opposite in order to give the widest possible spacing to reduce surface conductivity. The cylinders should be very close to equidistant. The apparatus should be dried for 2 hours at 100°C. before use.

Heat the sample to be tested to about 110°C. on a hot plate and pour into the electrode assembly until the level of the liquid is 1/4 to 1/2" above the electrodes. Care must be used to make sure that the

lip of the container, from which the liquid is poured, is clean. Tilt the beaker in such manner that all air bubbles in the Aroclor will rise to the surface.

Air cool to 103-104°C. then without removing thermometer, transfer to the oven which is maintained at 100°C. and connect the electrodes to the two flexible wires which lead to the megohm bridge. When the Aroclor comes to temperature (100°C.), remove thermometer, check centering of electrodes, and proceed to measurements.

Measurement with Megohm Bridge

Connect the two lead wires to the megohm bridge with the lead from the outer electrode to the LOW unknown post of the bridge; the inner electrode to the "+" unknown post. Connect the GROUND post on the left side to a water pipe. Swing the spring connector, pivoted on the "G" post, to the LOW post. Connect the attachment cord to the 110 V power supply.

With the Aroclor at 100°C., turn the control knob (CHECK-OPERATE-CHARGE) to the CHECK position. Throw all 3 switches at the rear of the Panel to ON. After 2 minutes bring the galvanometer pointer to zero by means of the ZERO ADJUST knob.

Then turn the control knob to CHARGE for one minute. Turn the knob to OPERATE and return the galvanometer pointer to zero by adjustment of the MULTIPLY BY switch and the megohm dial. Read after one minute. Repeat the CHARGE and OPERATE positions for verification.

The resistivity of the Aroclor is the product of the "multiply by" reading times the dial reading times the electrode constant. Report in units of 10^9 ohm cm, rounded off to two significant figures.

Example:

"Multiply by" reading: 100
Megohm dial reading 5.4 meg. ohms
Electrode Constant 815 cm

Resistivity = 100 X 5.4 X 815
= 4.4×10^5 meg. ohm cm
= 440×10^9 ohm cm

Values for resistivity are qualified by designation of temperature and voltage. These are 100°C. and 500 volts for the test above. Like conditions are used at Plant B.

METHOD: Resistance Measurements between Conductors

Care of Apparatus

The "Operating Instructions", General Radio Form 458-B, which accompany the megohm bridge, recite details of installation, measurements, uses, and construction, illustrated by figures and circuit and wiring diagrams. No attempt is made to reproduce such information here. The operator should acquaint himself with the contents of the G.R. manual and refer to it for maintenance of tubes and parts. When not in use the megohm bridge should be closed and stored in cabinet.

Cleaning Electrodes

After the measurements are made the electrodes are removed from the tested liquid, and allowed to drain until the liquid stops running from the electrodes. While still hot they are placed in a beaker filled with benzene under a well ventilated hood and allowed to cool. When cool, the electrodes are removed from the benzene and scrubbed with powdered tri-sodium phosphate and a benzene carbontetrachloride mixture (50-50 by volume). This scrubbing can be done with either a brush or the hands. The electrodes are then rinsed with acetone, followed by tap water and then distilled water. The electrodes should not be touched by the hands after the acetone wash. After final rinsing the electrodes are placed in an oven at 120°C. for one hour or until they are used again.

The glass spacer plates are cleaned and handled in the same manner as the electrodes except that they are wrapped in lense paper before being placed in the oven.

Calibration

For a rough check on the condition of the bridge, one or more cartridge-type resistances of known approximate value should be kept on hand for periodic or emergency verification of the meter itself.

rs
8/3/48

Monsanto Chemical Company

Anniston Method No. 14-56-52

METHOD: Cleaning Dielectric Testing Cells

The following method is currently in use in Monsanto laboratories, for cells for the dielectric testing of Aroclors, particularly the GE resistivity cells. All steps up to (7) are performed with the cell assembled and in the beaker used in tests.

(1) Drain cell while hot.

(2) Fill with hot trichlorobenzene (TCB); heat for 10-15 minutes.

(3) Discard TCB from (2), fill with unheated TCB; let stand a few minutes, drain.

(4) Rinse at least twice with methanol, and twice with tap water.

(5) Fill with hot 10 to 12% trisodium phosphate (TSP) solution, heat for 10-15 minutes, drain.

(6) Rinse thoroughly with tap water.

(7) Disassemble the cell, rinse the members thoroughly with distilled water. Dry at 120-130°C. for at least one hour.

(8) If cell is left in oven over 8 hours, it must be recleaned immediately before use.

NOTES

The TSP solution should be heated to about 100°. The TCB should be heated to about 100 to 140°.

Hot TCB is necessary to cut Aroclor 1260. Other solvents, e.g., benzene can be used on lower Aroclors. These suffer the handicap of flamability and lower boiling point.

The second filling of TCB, from (3), may be saved for re-use in (2), provided the sample in the previous test was not too badly contaminated.

The TSP solution should be made up fresh about every two days if in continuous use.

If a thermometer is to be used in stirring the Aroclor while heating, it should be cleaned by the method outlined above.

Developed by:
R. J. Good
9/24/52

DSW 331808

Monsanto Chemical Company

Anniston Method No. 14-36-48

SUBJECT: Dielectric Constant of Liquid Aroclor

METHOD: Measured at 100°C. and 1000 cycles

Scope

This method applies to mineral oils and oil substitute used for insulating purposes.

Apparatus

Capacitance Bridge	General Radio Co.,	Type 716-AM
Cathode Ray Null Detector	General Radio Co.,	Type 707-A
Oscillator	General Radio Co.,	Type 606-A
Constant Temperature Oven	Cenco-DeKhotinsky	#95050-A
~~Variable Air Condenser~~	~~General Radio Co.,~~	~~Type 334-F~~
1500 cc. Pyrex Beaker		

Procedure

Electrode and beaker must be cleaned as directed under "Cleaning Apparatus for Dielectric Constant", and be at 100°C.

Heat Aroclor in the ~~can~~ Beaker to 105°C. Pour over electrode ~~until plates and ceramic insulation~~ are covered. ~~Connect leads.~~ Stir with thermometer outside oven until temperature reaches 101°; ~~place in oven which has been carefully adjusted to 100°C~~. At the time sample is placed in oven its temperature should not have fallen below 90°C.

Connect leads to capacitance bridge, observing that the lead from insulated side of condenser goes to insulated terminal on bridge. Balance bridge carefully according to direction under "Balancing Capacitance Bridge". Until Aroclor and oven temperatures have become identical the bridge balance will constantly shift. After oven temperature reaches 100°C., maintain bridge balance until no pronounced shift is evident. Record reading of capacitance scale multiplied by multiplier setting.

$$\text{Dielectric Constant} = \frac{\text{Capacitance in Aroclor at 100°C.}}{\text{Capacitance in air at 100°C.}}$$

Balancing Capacitance Bridge

Condensed procedure. Operator should read operating instruction for Null Detector for complete details.

1. Connect to power supply leads to oscillator and null detector. Ground the oscillator.

2. Turn on oscillator by pushing 5000 ohm, 10 multiplier, 100 cycle frequency buttons. Adjust harmonic control until oscillation begins as shown by closing of sector in cathode ray tube.

DSW 331809

3. On null detector turn brilliance knob to extreme left, the focus knob to extreme right, gain control to extreme left, sweep amplitude at mid-scale position, the sweep frequency switch on line side.

4. Turn on power switch, lighting pilot light.

5. Wait fifteen seconds, turn brilliance knob to extreme right.

6. Adjust focus and brilliance knobs until sharp fine line.

7. Connect external terminals on detector to output terminals on oscillator. Switch sweep frequency to external side.

8. Connect detector terminals of bridge to input terminals of detector, taking care that the black wire (ground wire) of the lead goes to grounded terminal on both ends of lead.

9. Set gain control at mid-scale and selectivity to extreme left. Turn sweep amplitude to extreme left, obtaining vertical line. With

10. With turning control knob obtain the maximum length of this line, keeping it always under 1/4 inch with gain knob.

11. Set sweep amplitude to extreme left.

12. Set gain control to give vertical straight line of about 3/8 inch.

13. Balance bridge by adjusting power factor and capacitance dials until only a dot remains on the screen when the gain control is at extreme right.

14. Set sweep amplitude beyond mid-point, and slightly displace the power-factor dial to obtain a tilted elipse.

15. Adjust phase control until this ellipse closes into a straight line inclined to the horizontal.

16. Bring power-factor dial back to balance position, which should restore the horizontal straight line. Displace this control the same amount in the opposite direction. This should again tilt the straight line but in the opposite direction. Adjust phase control until there is no tendency for this line to open up when it is swung 30 degrees from horizontal in both directions.

17. Throughout these adjustments the other bridge control (the

capacitance Dial) must be in balance position. If the ellipse gradually opens when the line is horizontal it may be brought back to the line again by making slight changes in this control. If confusion results, balance both controls again as in (13) and repeat the succeeding adjustments.

This is the position of bridge balance: An alteration of the power-factor control will tilt the line which does not open appreciable thru an angle of 30° to the horizontal both ways; an alteration of the capacitance dial opens the ellipse but does not change the inclination of the major axis.

The apparatus may be left in this balanced condition, turned off by switching off first the brilliance knob of the detector, then the power switch, then turning off the oscillator.

For successive measurements the controls are not changed, the apparatus left in adjustment. Then the following steps define the procedure:

1. Connect all leads as previously directed including that from null detector to bridge.

2. Turn on oscillator by pushing 100 cycle frequency button.

3. Turn on null detector, wait 15 seconds then turn on cathode ray tube. Turn sweep amplitude to extreme left and adjust brilliance to obtain a fine vertical line on screen.

4. Bring this line to a dot by adjusting the two bridge controls. Initial adjustment is facilitated by having "Gain" control to left. When final adjustment is obtained this knob must be at extreme right.

5. This dot remaining unchanged shows condition of bridge balance.

Cleaning Apparatus for Dielectric Constant

The condenser is removed from the tested dielectric and allowed to drain while still in the oven at 100°C. The condenser is then placed, while still hot, in a beaker of benzene under a well ventilated hood and allowed to cool. It is then thoroughly washed with powdered trisodium phosphate and a 50-50 mixture of benzene and carbon tetrachloride followed by an acetone rinse. The operator should be very careful not to touch the metal plates of the condenser after this point. The condenser is then rinsed thoroughly with tap water followed by distilled water and dried on a clean paper in an oven at 120°C for at least one hour or until it is again used.

mwb 3/1/48

DSW 331811

Monsanto Chemical Company

Anniston Method No. 14-42-48

SUBJECT: Acid Number of Liquid Aroclors

METHOD: Titration with Alkali using Phenol Red

Scope

The Acid Number is the weight in milligrams of sodium hydroxide required to neutralize one gram of Aroclor to pH 7.6. It thus includes any hydrochloric acid, ferric chloride, and other inorganic or organic constituents having acid characteristics. It corresponds to the Neutralization Number of ASTM Designation D188 except that the latter is expressed in terms of KOH instead of NaOH and has a somewhat higher (phenolphthalein) end point.

Procedure

To 100 ml. of adjusted (pH 7.6) solvent containing indicator slowly add 0.1 N NaOH dropwise until the color corresponds approximately to pH 7.6 when compared against a suitable reference solution. Readjust if necessary to maintain pH 7.6. Add 50 grams (± 1 g.) of Liquid Aroclor and mix well. Titrate from micro buret with 0.01 N NaOH until the original red color of the solvent is restored. A portion of solvent in a similar container is a useful reference in determining the end point. The match is governed by estimation of the intensity of red because the hue is not exactly reproduced on account of the yellow color introduced by the Aroclor. 1 ml. of 0.01 N NaOH = 0.4 Mgm. NaOH. Calculate in terms of Mgm. NaOH per gm. of sample. Report to 4 decimal places.

$$\frac{\text{ml. of 0.01 N NaOH} \times 0.4}{\text{g. of sample}} = \text{Mgm. NaOH/gm.}$$

Example: If titration is 0.08 ml. for 50 g. of Aroclor, the Acid Number is 0.08 X 0.4/50 = 0.00064 Mgm. NaOH/gm. Report as 0.0006.

Solutions Required

Solvent: Mix 600 ml. of benzol, 200 ml. of 3-A alcohol and 200 ml. of acetone. Add 5 ml. of 0.2% phenol red solution.

Phenol Red Solution, 0.2%: To 0.1 gm. of dry indicator in a small beaker add exactly 2.0 ml. of 0.1409 N NaOH. Stir to dissolve and dilute with 3-A alcohol to 50 ml.

Reference Buffer pH 7.6: Dissolve 0.41 g. NaH_2PO_4 and 0.59 gm. Na_2HPO_4 (both anhydrous) in 100 ml. of water. Add 0.5 ml. of 0.2% phenol red. Solution is used for reference in adjusting the solvent if LaMotte pH 7.6 color standard is not available.

DSW 331812

Standard 0.01 N NaOH: 5 ml. of .5 N NaOH to 250 ml. vol. of 3-A alcohol. Protect at all times from atmospheric CO_2.

Note

By using 250 ml. acetone in solvent instead of 200 ml. a 100 gm. sample of 1254 may be taken for analysis.

FAB:cm
1-30-45

Copied by rs
4/23/48

Monsanto Chemical Company

Anniston Method No. 14-43-48

SUBJECT: Color of Aroclor on N.P.A. Scale

METHOD: Hellige Pocket Comparator

Scope

The method is applicable to Aroclors 4465, 5460, or others having N.P.A. colors in the range 1 to 5.

Procedure

Warm the Aroclor is necessary to obtain pouring consistency. Fill the test cup of the Hellige Pocket Comparator, Model 605, with Aroclor, insert cup in the comparator, and compare against the color disc (No. 620c-50, Lubricating Oils and Petrolatum) which has N.P.A. colors from 1 to 5 in one-half steps. Report color to nearest 1/4. Clean the test cup with a mixture of CCl_4 and benzol, with final rinse of benzol.

ASTM color numbers coincide with National Petroleum Association color number (1915). The relation of the N.P.A. color numbers to other color scales is tabulated below:

NPA Color Nos. (1915)	NPA Names	Union Petroleum Co.	ASTM Color Nos.	Lovibond Analysis Red 200	Yellow 510	Blue 1180
1/4	-	-	-	-	0.6	-
1/2	-	-	-	-	1.1	-
3/4	-	-	-	-	1.7	-
1	Lily white	G	1	0.12	2.4	-
1-1/2	Cream white	H	1.5	0.60	8.0	-
2	Extra pale	I	2	2.5	26.0	-
2-1/2	Extra lemon pale	J	2.5	4.6	27.0	-
3	Lemon pale	K	3	6.9	32.0	-
3 -1/2	Extra orange	L	3.5	9.4	45.0	-
4	Orange pale	M	4	14.0	50.0	.55
4-1/2	Pale	N	4.5	21.0	56.0	.55
5	Light Red	O	5	35.0	93.0	-
6	Dark Red	P	6	60.0	60.0	.55
7	Claret Red	Q	7	60.0	106.0	1.80
8	-	R	8	166.0	64.0	-

FAB:cm
1-26-45

Copied by rs
4/27/48

DSW 331814

Monsanto Chemical Company

Anniston Method No. 14-44-48

SUBJECT: Color of "Water White" Aroclors

METHOD: Comparison against A.P.H.A. Scale

Scope

The method is applicable to Aroclors 1254, 1260, or others having color less than N.P.A. #1 ("Water White").

A.P.H.A. Standards

These color standards are acid solutions of potassium chloroplatinate and cobaltous chloride. The unit of color is that produced by 1 mg. of platinum per liter. The ratio of cobalt to platinum may be varied if necessary to match the hue. Because the hue of Aroclor 1254 and 1260 seems best matched without cobalt, this has been omitted in the preparation described.

Dissolve 1.245 g. of potassium chloroplatinate (K_2PtCl_6) containing 0.5 g. of platinum in water with 100 ml. of concentrated hydrochloric acid, and dilute to 1 liter with distilled water. This solution has a color of 500.

Prepare standards from 10 to 100 in steps of 10 by diluting 1, 2, 3 ml. etc. of the above solution with distilled water to 50 ml. in standard high form Nessler tubes. Protect the tubes from evaporation.

Refer to page 13 of "Standard Methods for the Examination of Water and Sewage" eighth edition (1936) for details of standards containing cobalt. Published by American Public Health Association, 1790 Broadway, New York.

Procedure

Fill a matching Nessler tube with Aroclor to a height equal to that in the standard tubes. Compare with standards by looking vertically downward through the tubes upon a white or mirrored surface placed at such an angle that light is reflected upward through the column of liquid. A color tube support (Fisher #7-065, 50 ml.) is convenient.

Inasmuch as the proportions of the standard color solution in the comparison tubes are such as to represent an aliquot part of a liter, the readings are direct as parts per million. Colors up to 100 are recorded to the nearest 5. Direct comparisons are satisfactory up to 100. Above this the sample should be diluted with Carbon tetrachloride (maximum A.P.H.A. color, 5), and the color obtained to nearest 10 by multiplying by the dilution ratio.

FAB:am
1-30-45

DSW 331815

Copied by rs
4/27/48

Monsanto Chemical Company

Anniston Method No. 14-46-48

SUBJECT: Acid Number of Solid Aroclors and Heavy Liquid Aroclors

METHOD: Titration with Alkali using Phenolphthalein

Procedure

Dissolve 50 grams of the Aroclor in 50 ml. benzene in a 400 ml. beaker, warming to hasten solution. Add 50 ml. 3A alcohol, 200 ml. distilled water, heat to boiling, add 1/2 ml. 1% phenolphthalein solution, and titrate, in the case of the distilled Aroclors with 0.01 N sodium hydroxide solution, to a faint pink color which is permanent for at least two minutes after vigorous stirring. A reagent blank should be run concurrently. The acidity of the sample is calculated as milligrams of sodium hydroxide required per gram sample.

Calculations

Using 0.01 N NaOH, Acid No. = ml. titration X 0.4/sample weight
For 50 gm. Acid No. =(ml. tit. = ml. blank) X 0.008

Using 0.1 N NaOH, Acid No. = ml. titration X 4.0/sample weight
For 50 gm. Acid No. = (ml. tit. - ml. blank) X 0.08

Apparatus

10 ml. burette in .05 divisions.

Conversions

1. To obtain Neutralization Number in terms of KOH, multiply the Acid Number in terms of NaOH by 1.402. Inverse factor is 0.713.

2. Multiply the Acid Number by 1000 to obtain the parts of NaOH required to neutralize one million parts of Aroclor. Thus Acid Number of 0.0006 is equivalent to 0.6 parts of NaOH per million parts of Aroclor.

3. The large amount of water used causes sharper separation of the aqueous layer from the non-aqueous layer, thereby making the endpoint more easily distinguishable. If the titration is carried out against a very light background or in a fluorescent light, the end point is easily seen.

4. Aside from free acid as a cause for NaOH consumption, ferric chloride which is usually present in small amounts, also consumes NaOH in neutralization to phenolphthalein. It therefore follows that an Aroclor of very low acid number must be also extremely low in ferric chloride.

FAB:cm
1-26-45

DSW 331816

Copied by rs - 4/27/48

Monsanto Chemical Company

Anniston Method No. 14-47-48

SUBJECT: Pour Point of Liquid Aroclors

METHOD: A.S.T.M. D-97-39

The pour point of an Aroclor is the lowest temperature at which the material will flow when it is chilled under certain prescribed conditions listed in ASTM D 97-39.

Apparatus

Apparatus consists of test jar, thermometer, cork, jacket, disk, gasket, and bath as described under A.S.T.M. method D97-39. The thermometer is the A.S.T.M. Cloud and Pour Test, range -38 to 50°C. Present apparatus is the Emil Greiner Co. #GR 2234 single unit with Gr2242 test jar.

Procedure for Aroclor

Follow D97-39 in all respects except that the centigrade scale is substituted. Only an outline follows: Add 40 ml. of Aroclor to test jar. Adjust thermometer to center of jar with beginning of capillary 1/8 inch below surface of the Aroclor. Warm the Aroclor without stirring to 46°C. in a bath at 46-48°C. Cool to 32°C. in air or water bath at 25°C. Transfer the jar to the jacket of the cooling bath which is maintained at 0 to 3°C.

Beginning at a temperature 8 to 10 degrees above the pour point, at each interval of 2°C., remove the test jar from the jacket and tilt just enough to determine if there is movement of the Aroclor. This inspection should not require more than 3 seconds. When movement is not promptly apparent, the test jar should be held in a horizontal position for exactly 5 seconds as noted by a stop watch. If movement occurs, immediately return the test jar to the jacket.

Repeat test for flow at the next temperature 2°C. lower. Continue the test in steps of even degrees centigrade (14,12,10, etc.) until the Aroclor shows no movement when the test jar is held horizontally for exactly 5 seconds. This is the solid point.

The pour point is the previous temperature, 2°C. above the solid point. The pour point is thus the lowest temperature at which the liquid will flow or pour under the test conditions.

Technicians must familiarize themselves with D97-39 which gives complete details of apparatus and procedure, reproducibility of results, effects of thermal history, etc. The directions above are inadequate except as a working outline.

The bath temperature is controlled by adding dry ice to acetone for pour points below 0°C.

DSW 331817

FAB:cm - 1-30-45
Copied by rs - 4/27/48

Monsanto Chemical Company

Anniston Method No. 14-48-48

SUBJECT: The Testing of Aroclors for Inorganic Chlorides

METHOD:

Solutions Required

Standard Chloride Solution: Dissolve 0.660 grams of pure sodium chloride in sufficient water to measure exactly 500 ml.; 1 ml. then contains 800 micrograms Cl. Pipette 25 ml. of this solution to a 500 ml. flask and make to the mark to give a 0.00113 M NaCl solution, 1 ml. of which contains 40 micrograms of Cl. Preserve in a Pyrex bottle. Caution: The water used in preparation must be chloride free by the Tyndall Beam test; the 0.00113 M solution should not be kept over 1 month.

Silver Nitrate Solution 10%: Dissolve 5 grams of silver nitrate crystals in 40 ml. of water and add 10 ml. of concentrated nitric acid.

Chloride free distilled water.

Apparatus Required

LaMotte 10 ml. color tubes (13 mm. X 100 mm).

Test tube rack with a black surface at the base for uniform comparison (Note: we are using black gasket rubber over the entire board that supports the base of test tubes during comparison.)

Box. About 7" long and 4" wide. This box contains holes in top and notches in the bottom to hold Lamotte color tubes. It is just high enough to allow 1/4" of test tube to extend through the holes in the top. The box should be painted a flat black on the inside and on top and should contain enough holes to accomodate at least seven tubes.

Method

Clean all flasks, tubes, and pipettes to be used. Then rinse with dilute nitric acid then with chloride free distilled water.

Add 50 ml. chloride free water to a 250 ml. Erlenmeyer flask, heat to boiling, add 200 gram of the Aroclor to be tested, which has been heated to 100°C, shake vigorously for 1 minute, cool in cooling pan. Carefully decant some of the water extract to a separatory funnel. Wash with pure ethyl ether (chloride free).

Transfer 10 ml. of the ether washed extract to a 10 ml. LaMotte color tube after rinsing tube with portion of extract. Prepare standards by diluting exactly 5 ml. of the 0.00133 M NaCl solution to 100 ml. in a

DSW 331818

volumetric flask. One ml. then contains 2 micrograms of Cl. Transfer 1, 2, and 3 ml. portions to 10 ml. tubes. Make to the 10 ml. mark with Cl free H_2O. Place standards and sample in light free box from which only the top of tubes are exposed add 0.5 ml. of the silver nitrate to each tube compare in a specially prepared test tube rack.

The comparison is to be made quickly and from a constant source of light, preferably day light from a window away from the sunlight. Comparison is easier when other sources of light are shielded from the comparison rack. Look directly into the top of the tubes and compare. Note the known solution which most nearly matches the water extract estimating to the nearest whole microgram. The extract blank and the distilled water should show no turbidity.

Calculation

Divide the chloride content in micrograms of the known solution which matches the extract by the weight of the Aroclor represented by the extract to obtain the chloride content of the Aroclor in p.p.m.

In the procedure described, the 10 ml. extract represents 40 grams of Aroclor (1/5 of 200 g. taken). The three knowns contain 2, 4, and 6 micrograms of Cl, thus representing 0.05, 0.10, and 0.15 p.p.m. respectively. Values should be reported to the nearest 0.025 ppm.

Precautions

Because of the sensitivity of this test, serious errors may result through very slight contamination or inattention to details. All equipment must be scrupulously cleansed, the distilled water absolutely chloride free and testing done away from fumes of HCl or chlorine fumes.

rs
4/27/48

Monsanto Chemical Company

Anniston Method No. 14-53-48

SUBJECT: Water Content of Liquid Aroclors

METHOD: Titration with Karl Fischer Reagent

Outline

The Aroclor is dissolved in a mixture of benzene and methanol which has been titrated with Fischer reagent to the characteristic end point. Fischer reagent is a solution of iodine, sulfur dioxide, and pyridine. Iodine is consumed as long as any water is present. The solution is again titrated to obtain the water content introduced by the Aroclor.

Solutions and Apparatus

Karl Fischer Reagent: Weigh into a flask 264 grams of pyridine, Barrett 2-A or Eastman 214-H, and add 61 grams of liquid or gaseous sulfur dioxide. Add the liquid sulfur dioxide directly from the inverted cylinder by attaching to the outlet valve a glass tube extending into the pyridine. Sulfur dioxide gas can be bubbled into the pyridine until proper amount is added. Store in a glass stoppered bottle.

Transfer 65.6 grams of the pyridine-SO^2 mixture and 134 ml of absolute methanol to a 1 liter Florence flask. Cool thoroughly in an ice water bath. Add 16.9 grams of iodine, stopper, cool again before shaking. Alternately cool and swirl until the iodine is in solution. Makes 200 ml of reagent. Store in glass stoppered bottle. Let stand 24 hours before using. When fresh, one ml of reagent is equivalent to about 0.0033 gms. of water.

Aroclor Solvent: Mix 2 parts dry benzol with 1 part anhydrous methanol. The dry benzol is prepared by shaking the commercial grade with anhydrous calcium chloride, decanting into a flask and distilling, rejecting the first 10% to come over. Keep in glass stoppered bottles with minimum exposure to air.

Micro Buret: 10 ml. x 0.05 ml. Koch automatic with glass stoppered reservoir. Eck and Krebs #2460. Equip reservoir with a moisture guard tube containing Drierite or a similar dehydrating agent.

Procedure

Heat a clean 500 ml. narrow mouth Erlenmeyer flask in an oven or on hot plate until thoroughly dry. Sweep out for 10 minutes or until cool with air which has been thoroughly dried by passing thru tubes containing 8 mesh Drierite. Disengage the flask from the aspirating train and immediately close with a paper cap held by a rubber band.

DSW 331820

Without delay, transfer 100 ml. of the benzene-methanol solvent to the flask thru a small hole punctured into the paper cap. Cover again with paper. Titrate at once with Karl Fischer reagent to distinct red-brown end point which does not fade after swirling several times. The Fischer reagent is dispensed from the microburet the tip of which projects into the flask thru the puncture in the paper cap. At the end point any water content of the solvent has been reacted. Superimpose a fresh paper cap immediately.

Obtain weight of the solvent and well-covered flask on a Torsion balance or equivalent. Quickly introduce with a dry pipette about 100 grams of the slightly warmed Aroclor to be tested. Cover at once and weigh again to obtain the weight of Aroclor to nearest gram. Shake until the Aroclor is dissolved.

If the mixture is cloudy, warm slightly. If cloudiness persists another mixture should be prepared. Use less sample

Puncture a small hole in the paper cap for the insertion of the buret tip and titrate with Fischer reagent to the first definite brown color, agitating with a gentle swirling motion. The end point should persist through several swirls. The end point may slowly fade through slow access of atmospheric moisture in which case it is restored by a drop or two of reagent. If not so restored, the end point may have been a false one or the closure is faulty. A definite end point is impossible if humid air is not well excluded.

From the amount of reagent required and the weight of Aroclor used, calculate the water content in p.p.m.

Calibration of Fischer Reagent and Standard Water Solution

Standard water solution in methanol: Transfer exactly 0.40 ml. of water from a micro buret or a 1 ml. measuring pipet to a 100 ml. volumetric flask. Fill to the mark at once with anhydrous methanol and mix. One ml. of solution then contains 4000 micrograms of water in addition to any water which may be present in the methanol.

Transfer exactly 5 ml. of the above standard water solution (equivalent to 0.02 grams of water) to a 250 ml. Erlenmeyer flask which has been dried and swept with dry air. Close with paper cap and titrate with Fischer reagent in the same manner as the sample. The end point is sharp. A methanol blank, found by titrating 5 ml. of the methanol used in making up the standard water solution to a like end point, must always be subtracted from this titration.

Divide the amount of water taken by the net Fischer reagent required to obtain the titer of the Fischer reagent in terms of water.

Example: A 5 ml. aliquot of standard solution, equivalent to 20,000 micrograms of water, required 6.61 ml. of Fischer reagent while 5 ml.

of the methanol required 0.55 of Fischer reagent.

$$20,000/(6.61 - 0.55) = 20,000/6.06$$
$$= 3300 \text{ micrograms water/ml.}$$

Recalibrations: Because the Fischer reagent deteriorates rapidly, its titer must be determined for each days use. If the same standard water solution is used in recalibration as in the initial calibration, the titer of the Fischer reagent must be calculated from its total water content (including that introduced by the methanol). The total water is the product of the initial gross titration and its titer. This in the example above the total water content of a 5 ml. aliquot is 6.61 X 3300 = 21813 micrograms. If on recalibration 7.37 ml. of Fischer reagent is 21813/7.37 = 2960 micrograms of water per ml.

Two or more calibrations should be made with agreement within 0.025 ml.

Calculation of H_2O sample

Standardization of Standard H_2O sol.

Titrate 5 ml. mixture H_2O sol.
Titrate 5 ml. of methanol
Tit of 5 ml. mixture = A
Tit of 5 ml. methanol = B

A - B = ml. reagent for H_2O added = 5/100 of .4
Divide 5/100 X .4 by A - B = factor grams of H_2O/ml.
Multiply factor X B giving gms. of H_2O in 5 ml. of methanol.
Add this wt. of H_2O to .02 already added giving H_2O in 5 ml. (Total)

$$\frac{\text{Factor}}{H_2O \text{ St'd Titration}} = T_1$$

$$T_1 \text{ X sample Titration} = T_2$$

$$\frac{T_2 \text{ X } 1,000,000}{\text{wt. sample}} = \text{ppm } H_2O$$

FAB:cm
1-27-45

Copied by rs
4/27/48

DSW 331822

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

Copied by mwb
4/2/48

DSW 331823

I-A-d-B-4000

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.

Copied by rs
4/2/48

DSW 331824

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

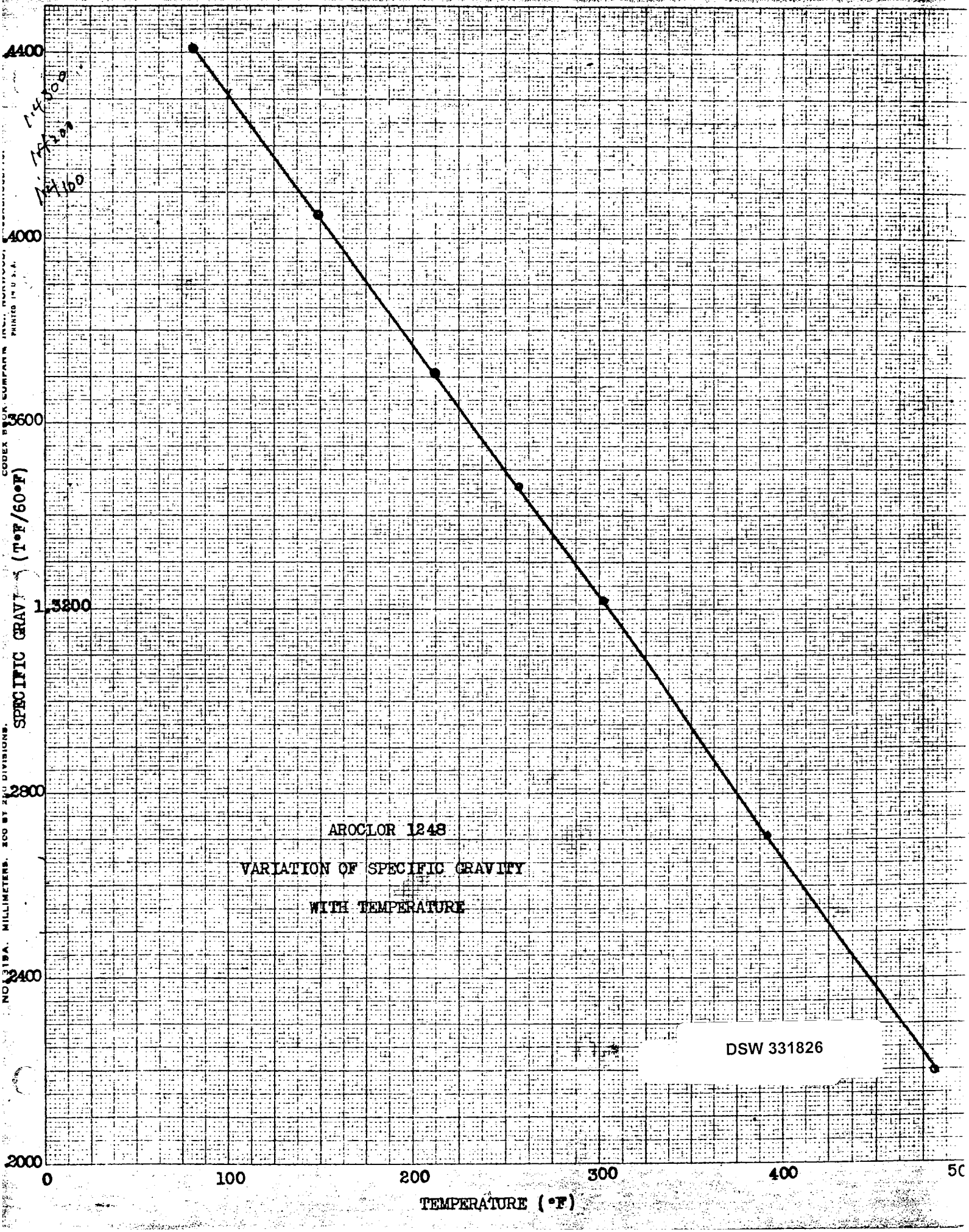
Letter - W.C.M./J.M. Cole, Tech. Rep.
2/16/48
Zyrox 3007 (11-308)

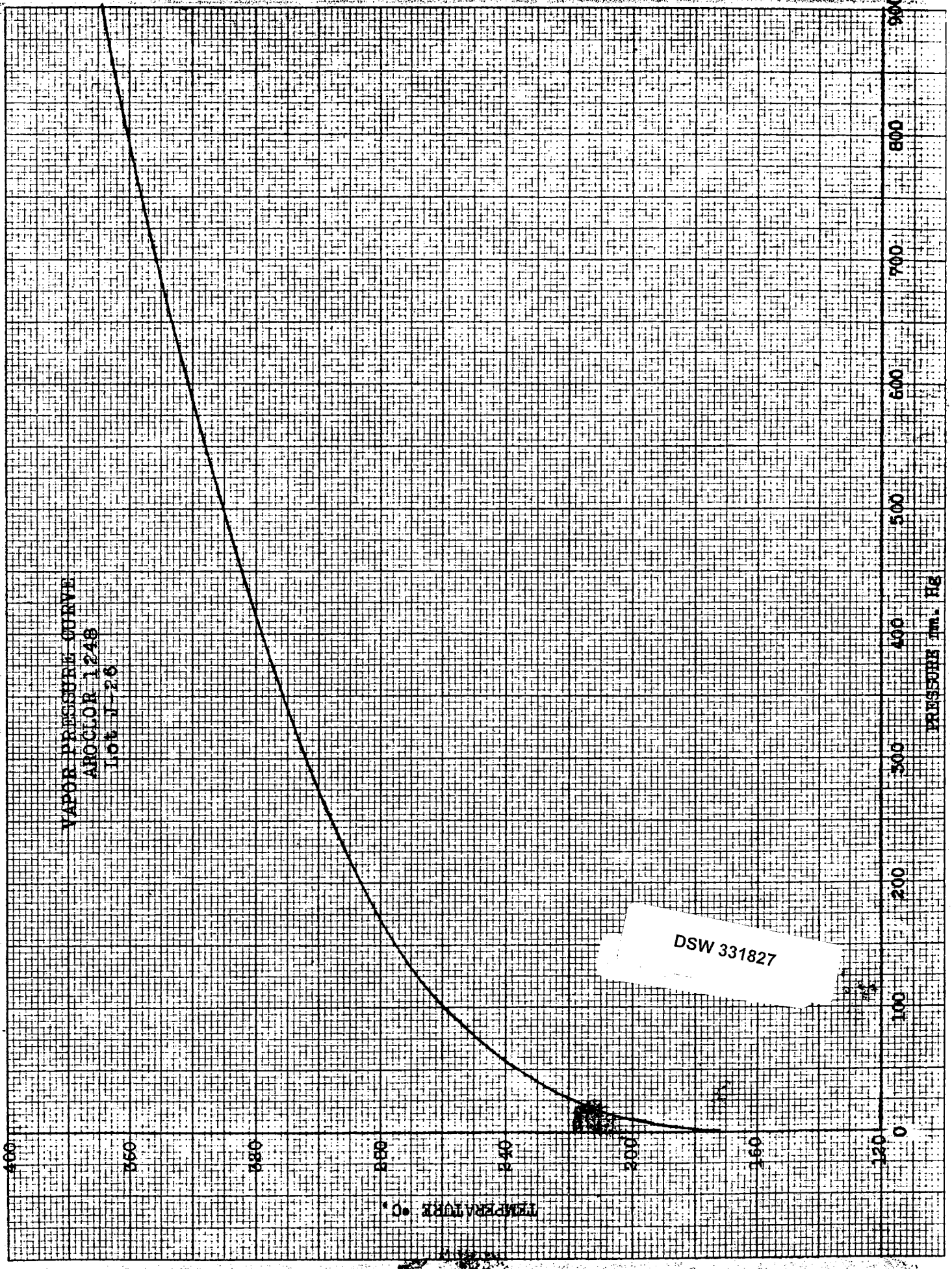
GENERAL INFORMATION

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.

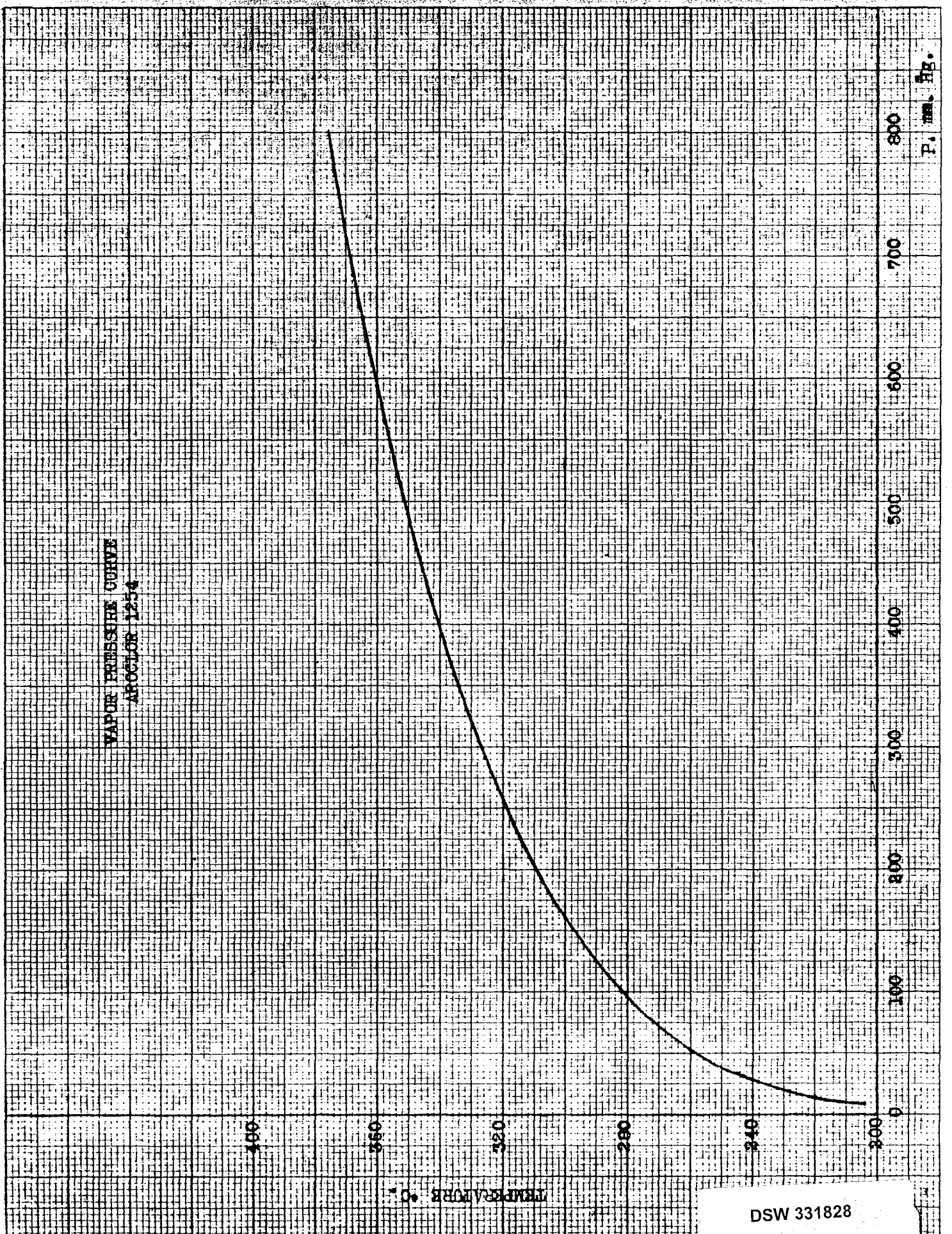
Copied by rs
4/2/48

DSW 331825




VAPOR PRESSURE CURVE
AROCLOR 1248
Lot 5-26
PRESSURE mm. Hg
TEMPERATURE °C.
900
800
700
600
500
400
300
200
100
0
400
360
320
280
240
200
160
120

DSW 331827


VAPOR PRESSURE CURVE
AROCLOR 1254
TEMPERATURE °C.
P. m. Hg.
0
100
200
300
400
500
600
700
800
300
320
340
360
380
400

DSW 331828

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 331829

STLCOPCB407840

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	4.33
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

Copied by rs
2/2/48

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

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".

Copied by rs
4/2/48

TB - 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.

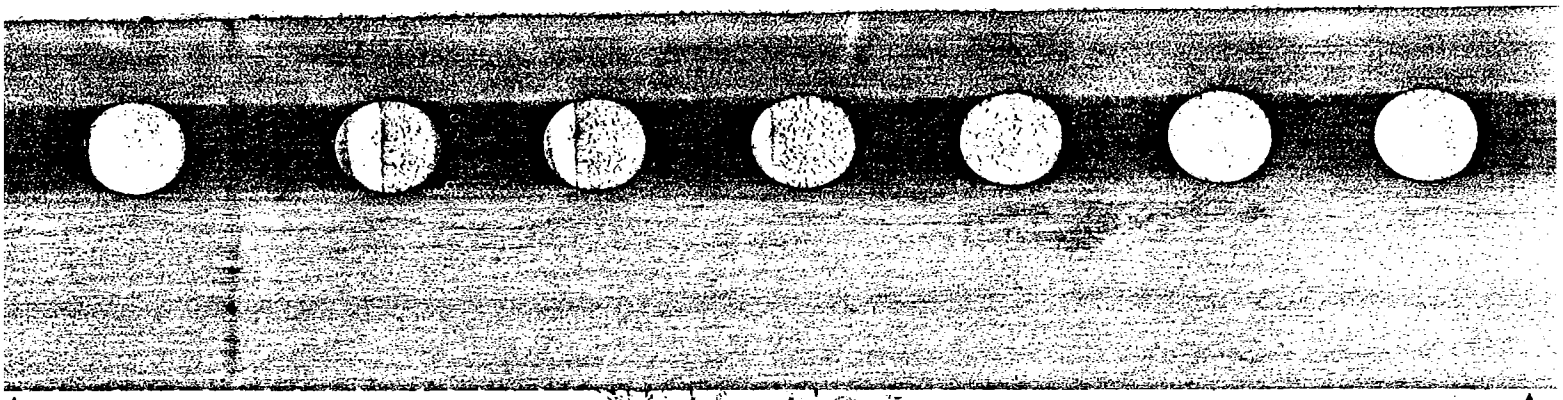
The log resistivity versus 1/T graph is useful as an indication of the change that occurs in resistivity with temperature - or what is related, the change in viscosity with temperature. The actual slopes of 4465 and 5442 cannot be assured since there should have been more measurements made at high temperatures.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

acm/
Copied by rs
1/15/48

DSW 331834



AN 4 44 CDT 6/13/55 AXXX-SL 241

W B DUNLAP

ANNISTON

DATA ON AROCLOR 1242 FRXX FOUND AFTER TALKING WITH YOU.

RESISTIVITY 50 DEGREE C. 9200 X 109

100 DEGREE C. 2400 X 109

150 DEGREE C. 1800 X 109

A M ELLENBURG

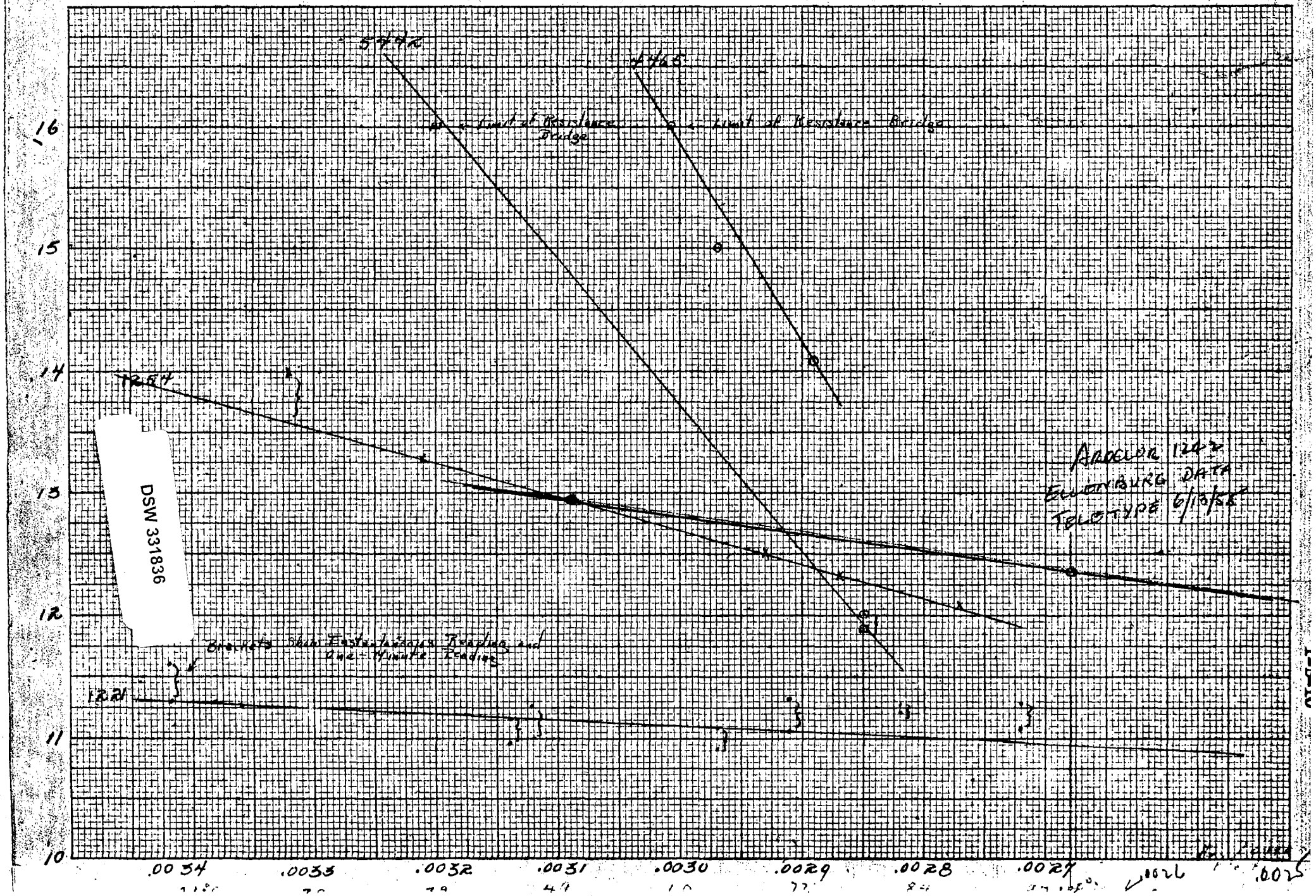
MONSANTO

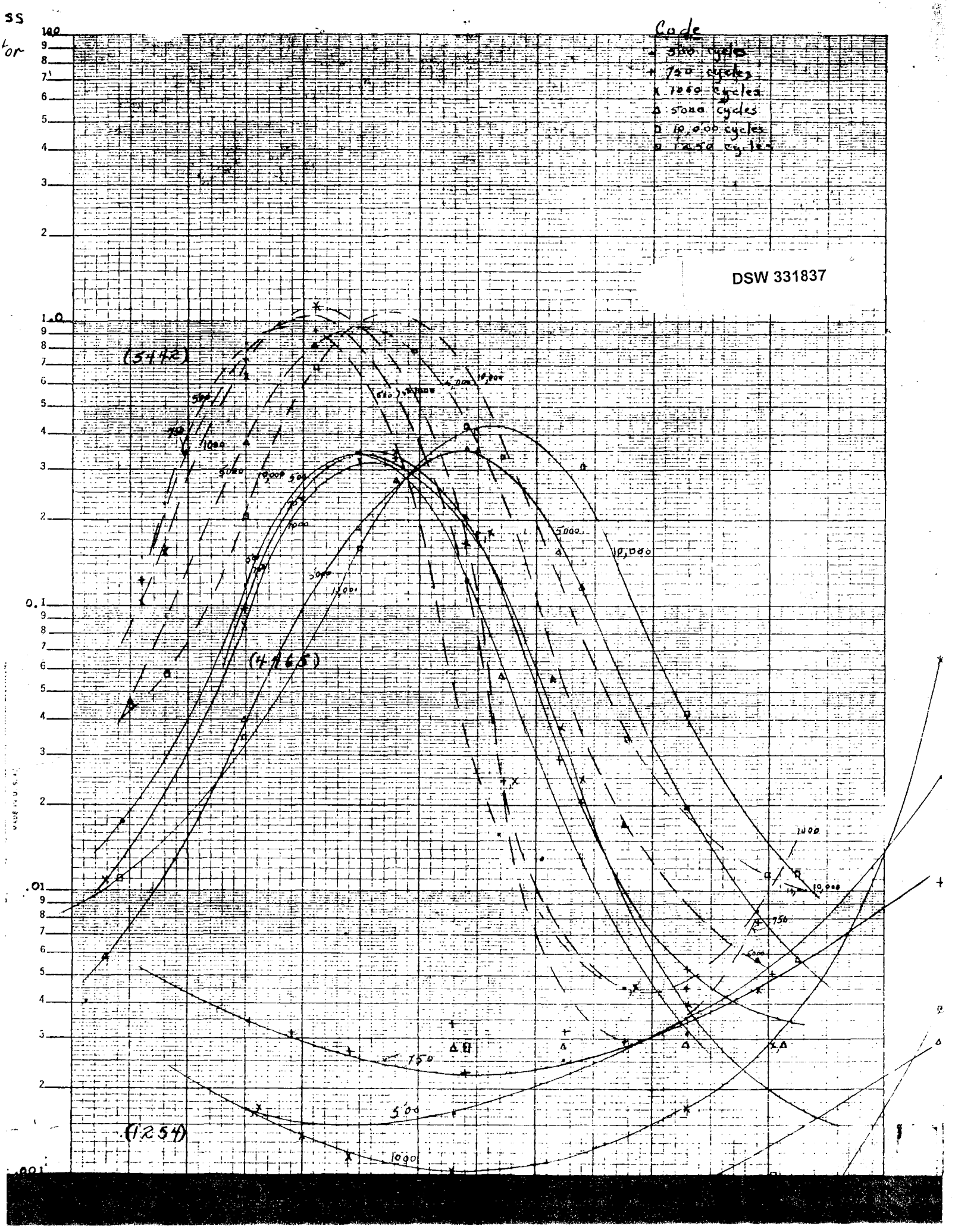
ST LOUIS

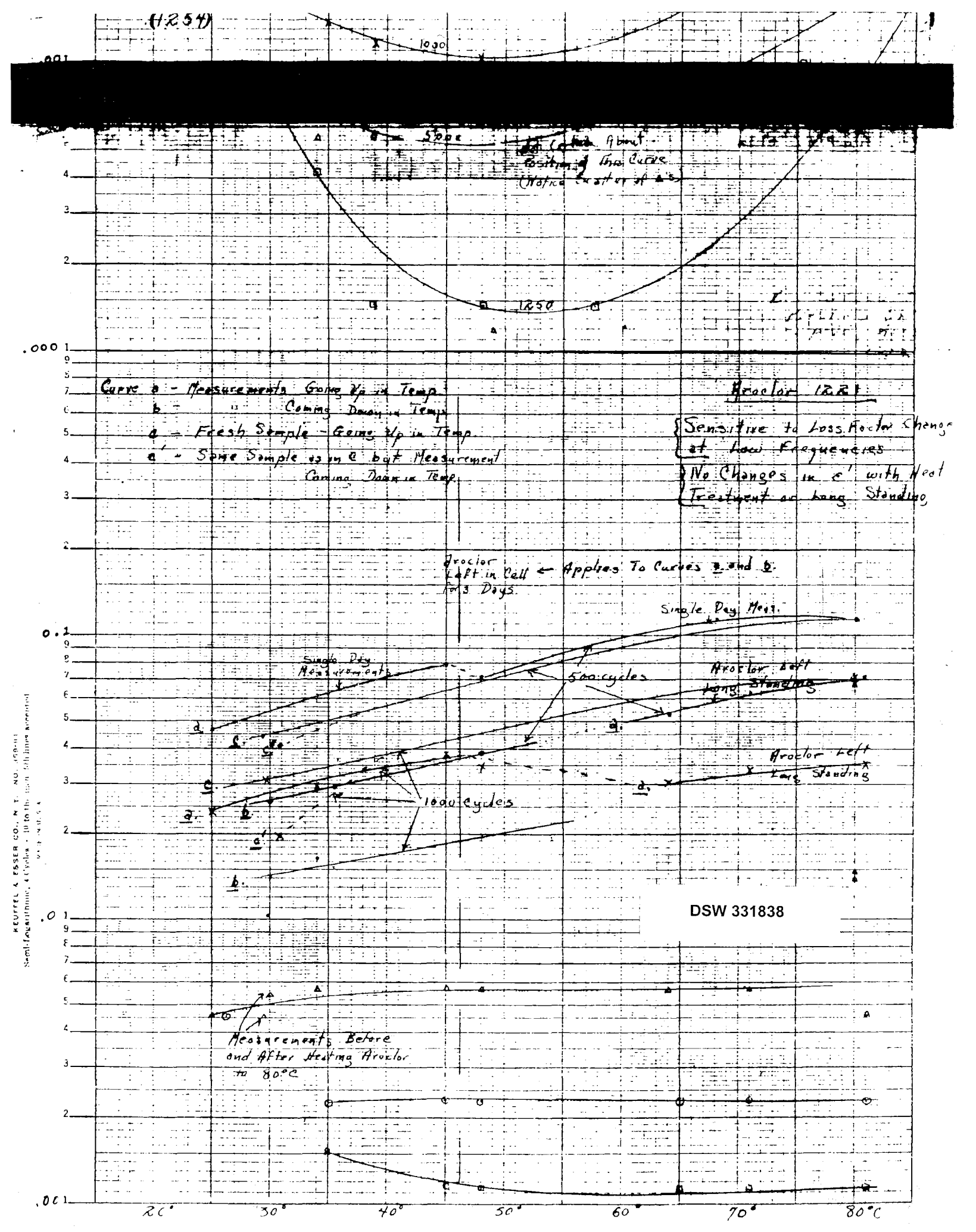
DSW 331835

KEUFFEL & ESSER CO., N. Y. NO. 359-11
10 × 10 to the ½ inch, 5th lines accented.
Engraving 7 × 10 in.
MADE IN U. S. A.

Log_{10} Resistivity

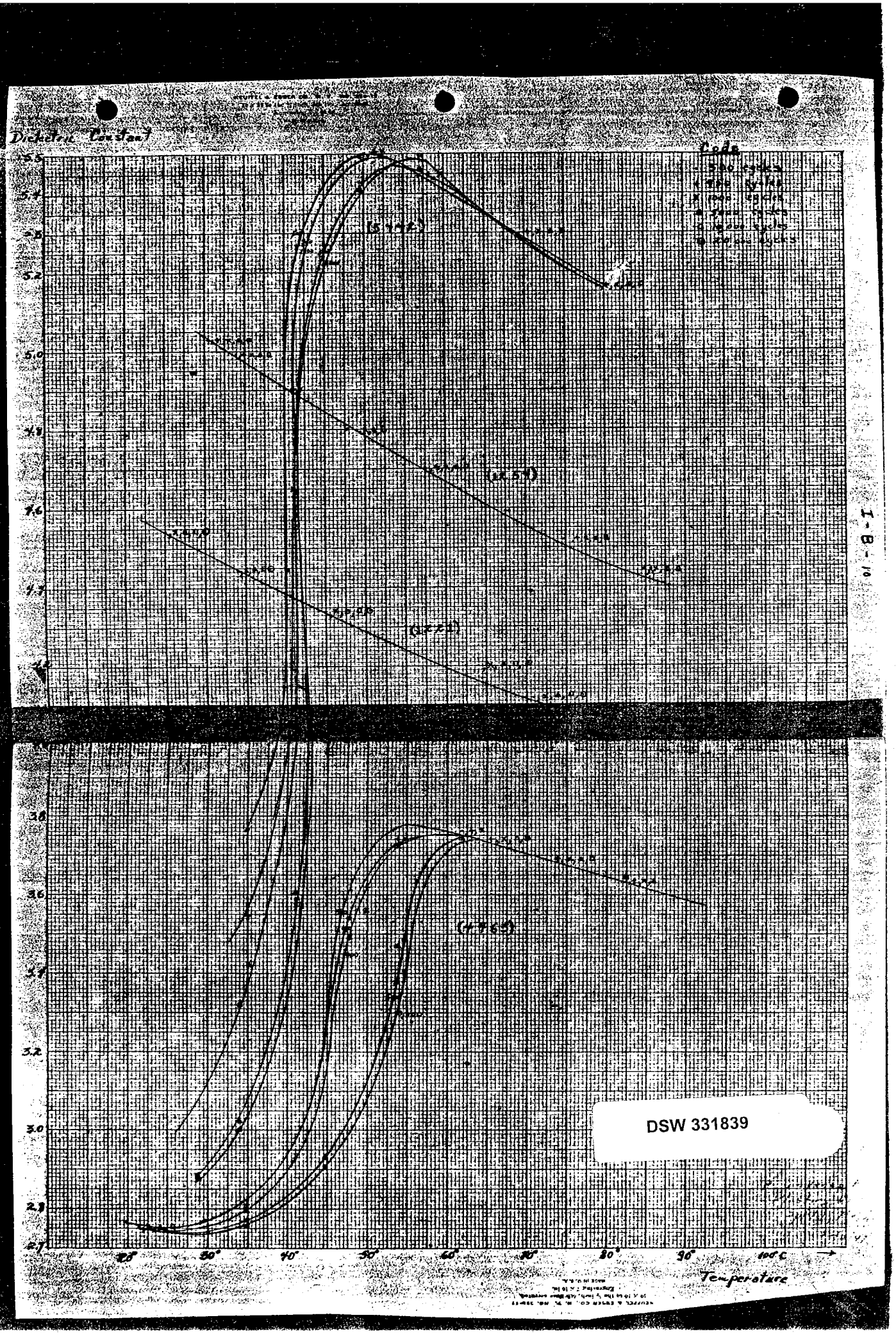






DSW 331838

STLCOPCB4078413



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:-

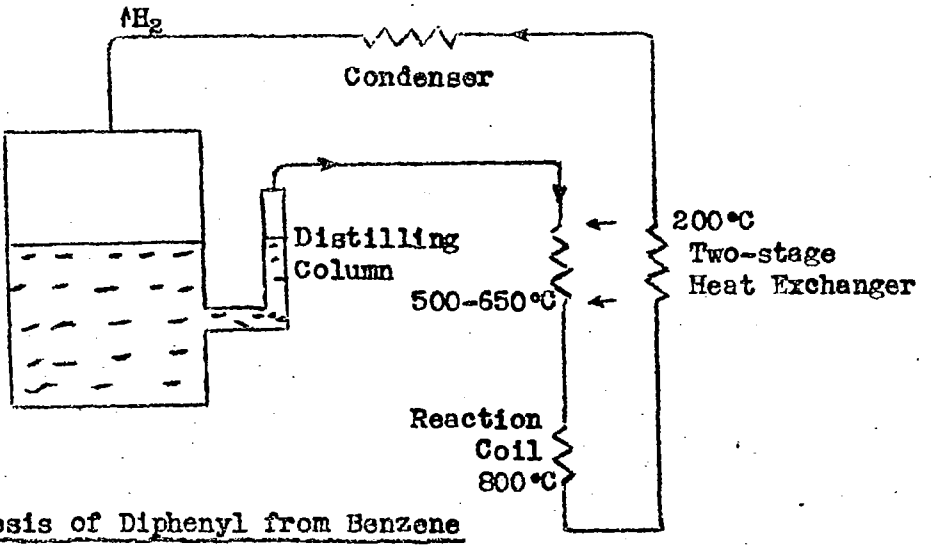
Siemens-Schuckert, Berlin.
A.E.G. (Hydrawerk), Berlin.
Micafil (Brown Boveri), Zurich, Switzerland.
Alsthom, Paris.
Ducati, Milan, Italy.

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 331841

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.

rs
11/19/47

III-A-b - 500

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.

rs
11/24/47

DSW 331845

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.

rs
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

rh

Copied by rs
2/2/48

DSW 331848

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.

Copied by rs
4/2/48

DSW 331849

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.

rs
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 "SCHUTZEL-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.

rs
2/3/48

January 27, 1949

Distribution:

1. Research File
2. R.L. Jenkins - C.B. Durgin
3. J.L. Christian - R.S. Weatherly
4. W.T. Durrett - F.P. LaBelle
5. H.F. Weaver
6. C.A. Hochwalt
7. E.P. Rucker
8. Edgar E. Hardy
9. J.F. Reeves
10. J.F. Reeves
11. A.M. Ellenburg
12. R.R. Knight
13. Paul Logue
14. P.G. Benignus

This Copy For → 15.

16.

17.

18.

Please insert the attached data in your copy of Report No. 2215, "Aroclor Data Book", issued April 27, 1948 by R. R. Knight, Research Department, Phosphate Division.

A. M. Ellenburg

AME:rs

DSW 331855

I-i-a-200

From: Plastics Research Report No. [illegible]

Final Report on "Evaluation of Monsanto Materials as Plasticizers".

Job No. 589.

AROCLOR 1232

Calculated molecular weight: 223
Refractive index*: 1.620 at 30°C.
Saponification equivalent*: none
Crystallization point*: -32.5°C.
Boiling point*: 290-325°C. at 760 mm.
Acidity*: .01 milligrams KOH/gm.
Acidity after heat: none
Color (Gardner Standard): 0
Color after heat: 0
Color after light: 11-12
Specific gravity*: 1.2625 at 25/25°C.
Water solubility: .0016% at 25°C.
Stability to hydrolysis*: essentially none
Odor: chloroaromatic
Viscosity: 25°C. - 6.24 centipoise
0°C. - 65 centipoise
-20°C. - 627 centipoise
Solubility: Soluble in: octyl alcohol, acetone, ethyl acetate, benzene, Skellysolve C, Skellysolve H, turpentine, castor oil, cotton seed oil.
Partially soluble in: methanol, ethanol, glycerol, linseed oil.
Insoluble in: glycol

rs
8-20-48

DSW 331856

TABLE I

DENSITY AND SPECIFIC VOLUME OF AROCLORS

AROCLOR 1242

Temperature (°C.)	Density (g./ml.)	Specific Volume (ml./g.)
49.9	1.3546	0.7382
102.2	1.3051	0.7662
150.3	1.2587	0.7945
201.7	1.2080	0.8278
254.0	1.1549	0.8659
298.2	1.1071	0.9033

AROCLOR 1248

Temperature (°C.)	Density (g./ml.)	Specific Volume (ml./g.)
25.1	1.4439	0.6926
49.9	1.4192	0.7046
103.3	1.3673	0.7314
150.1	1.3210	0.7570
201.7	1.2691	0.7880
254.0	1.2148	0.8232
298.2	1.1664	0.8573

AROCLOR 1254

Temperature (°C.)	Density (g./ml.)	Specific Volume (ml./g.)
25.1	1.5392	0.6497
49.9	1.5139	0.6605
102.9	1.4607	0.6846
150.1	1.4127	0.7079
201.8	1.3595	0.7356
205.5	1.3096	0.7636
298.2	1.2546	0.7971

TABLE I Cont'd

DENSITY AND SPECIFIC VOLUME OF AROCLORS

AROCLOR 1260

Temperature (°C.)	Density (g./ml.)	Specific Volume (ml./g.)
49.2	1.5956	0.6267
102.8	1.5424	0.6483
150.2	1.4931	0.6698
201.9	1.4386	0.6951
251.0	1.3854	0.7218
298.3	1.3322	0.7506

D. Ward
Central Research Department
December 21, 1948

Copied by rs
1-20-49

VISCOSITY - PRESSURE RELATIONSHIPS OF OILS AND CHLORINATED DIPHENYLS

R. B. Dow, M. R. Fenske and H. P. Morgan
The Pennsylvania State College

Industrial and Engineering Chemistry
Vol. 29, 1076-80, (1937)

The following data are taken from tables in the article and should be viewed critically. Specific heat data are out of line with our Aroclor figures.

TABLE I

Aroclor	Sp. Ht. Cal./g/C°	Centipoises Visc. 30°C.	Visc. 75°C.	Viscosity Index	Density 30°C.
1248	0.33	129	8.7	-334	1.445
1254	1.48	2740	24	-1989	1.540

Observations were made that the increase in viscosity with pressure was very great with the Aroclors and Aroclor 1254 exhibit one of the highest highest ratio of increase known for an oil.

TABLE II

VISCOSITY VARIATION WITH PRESSURE

Pressure psi.	30°C		75°C	
	1248	1254	1248	1254
14.2	129 cps	2740 cps	8.7 cps	24 cps
500	—	3140	—	—
1000	153	4020	9.3	25
1500	—	5670	—	—
2000	181	8740	10	26
2500	—	14300	—	—
3000	223	23600	11	29
3500	—	40500	—	—
4000	287	-----	12	33
5000	384	-----	13	40
6000	553	-----	15	48
7000	855	-----	17	59
8000	1270	-----	19	73
9000	1950	-----	21	92
100000	2940	-----	24	117
12,000			30	198
14,000			39	345
16,000			53	642
18,000			75	1290
20,000			111	2580
22,000			176	
24,000			300	
26,000			494	
28,000			816	
30,000			1350	
32,000			2196	

Copied by rs
7-12-48

TABLE II

VISCOSITY OF AROCLORS

AROCLOR 1248

Temperature (°C.)	Kinematic Viscosity (Centistokes)	Absolute Viscosity (Centipoises)
24.80	151.0	218.1
25.10	144.7	208.9
25.41	140.2	202.4
49.9	18.27	25.93
99.4	3.04	4.17
150.2	1.257	1.660
200.4	0.777	0.987
251.8	0.533	0.649
297.7	0.412	0.482

AROCLOR 1254

Temperature (°C.)	Kinematic Viscosity (Centistokes)	Absolute Viscosity (Centipoises)
24.80	585x10	901x10
25.41	505x10	777x10
49.9	96.7	146.4
50.1	95.9	144.9
98.7	5.86	8.59
150.6	1.785	2.521
200.4	0.991	1.349
250.3	0.658	0.863
251.8	0.652	0.853
297.6	0.485	0.608

D. Ward
Central Research Department
December 21, 1948

Copied by rs
1-20-49

DSW 331861

NON-FLAMMABLE DIELECTRIC ORGANIC COMPOUNDS

F. M. Clark - General Electric Company

Industrial and Engineering Chemistry
Vol. 29, 698, (1937)

Characteristics of Dielectric Liquid Compounds

Property	Pentachlor-Biphenyl	Pentachlor diphenyl Oxide	Pentachlor diphenyl Ketone	Hexachloro diphenyl-Methane	Trichlor Benzene
Burn Point	N.F.	N.F.	N.F.	N.F.	N.F.
Sp. Gr. (°C/15.5°C.)	1.51 (65)	1.59 (100)	1.43 (100)	1.52 (100)	1.46 (25)
Viscosity, SUS, °C.	45 (98)	590 (37.8)	54 (100)	84 (100)	50 (37.8)
Pour Point, °C.	+ 10	0	+ 15	+ 20	0-10 F.P.
Refr. Index (25°C.)	1.6580	1.6220	1.6370	1.6370	1.5700
Dielectric Strength, Kv.	40	40	40	40	40
Dielectric Constant (25°C.)	5.1	5.0	8.0	4.3	4.8
Arc formed Gases	N.E.	N.E.	N.E.	N.E.	N.E.
Sludging	None	None	None	None	None
Free Acid	None	None	None	None	None
Distillation Range, °C	360-400	230-250 (15 mm)	250-300 (25 mm)	290-340 (25 mm)	200-220

N.F. - Non-flammable.
N.E. - Non-explosive.

Aroclor 1254
Viscosity - McMicheal - Centipoises

Temperature - °C.	Viscosity - cps.
28	6000
31	3000
40	700
50	180
60	70
70	38
80	26
90	19
100	17

Copied by rs
7-12-48

DSW 331862

III-G-100

St. Louis

May 25, 1948

Messrs.
Edgar E. Hardy - Anniston
A.M. Ellenburg - Anniston
Paul Logue
T.H. Wheelock

Dr. R. L. Jenkins
Mr. C. B. Durgin
Anniston

Aroclor--Silastic Facts

Attached are two copies of the No. 5 series about Silastic Facts.

Recently Dow Corning submitted the following information to us:

"Silastic is swollen quite badly when subjected to most organic solvents such as toluene, benzene, etc. However, our laboratory has recently submitted a report concerning the effects of chlorinated diphenyl on Silastic. Silastic immersed in chlorinated diphenyl for seventy hours at 150°C. The change in the physical properties were noted and the figures indicated that Silastic is remarkably resistant to deteriorations from contact with the hot chlorinated diphenyl. The changes in the properties are similar to those produced by hot lubricating oils and can be summed up as follows:

(1) Lowering of the hardness by about 20 points.
(2) Improvement in the elasticity by several points.
(3) Practically no change in tensile strength.
(4) An appreciable increase in the ultimate elongation."

Western Felt Manufacturing Company at Chicago, Illinois, quoted Silicone 180 in sheet form as follows:

8x8" sheet, 1/4" thick	$11.25
20x20" sheet, 1/4" thick	63.50
24x24" sheet, 1/4" thick	89.50

Apparently the greatest drawback to the use of Silicone as a gasket material for hot Aroclors is the high price of the plastics.

Benignus

rh
encs

Copied by rs
9-24-48

DSW 331863

HANDLING AROCLORS

Gaskets - Aroclor and Pyranol

Letter F. B. Zienty to H. M. Hitchens - 9-20-48

"At a recent meeting it was stated by Dr. Suits of General Electric that silicone rubber gaskets were found to hold Pyranol effectively under conditions where other gasket materials gave poor performance with respect to freedom from leaks."

SALES DEVELOPMENT REPORT

P. G. Benignus - 2-18-48

Precision Die Casting Company, Cleveland, Ohio

"Ball-bearing swing joints made by Chiksan Tool Company at Brea, California used on flexible connections with Aroclor installations.

"G. E. Glyptal # 2 Red is excellent for sealing fluid on application to threaded pipe."

rs
9-24-48

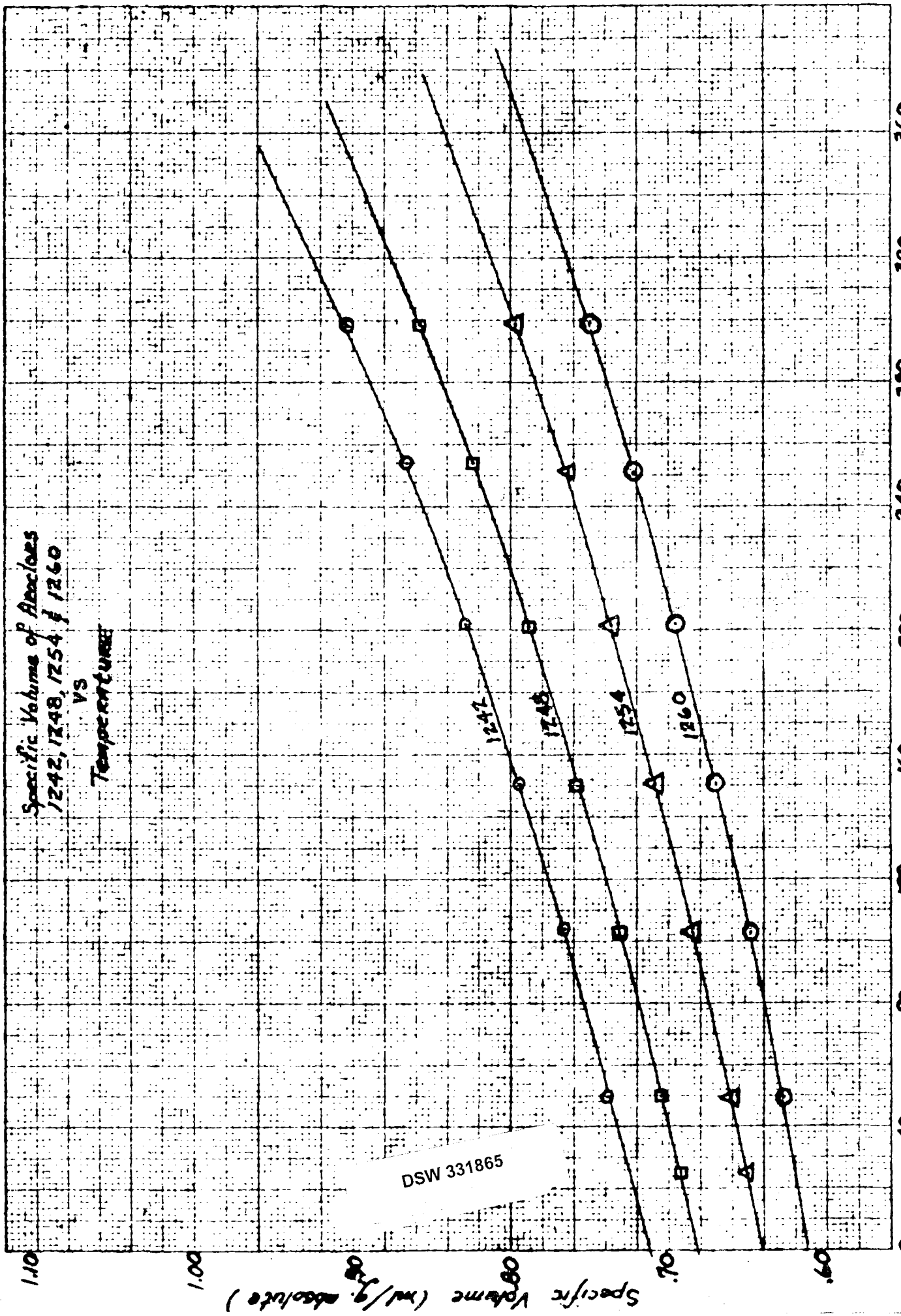
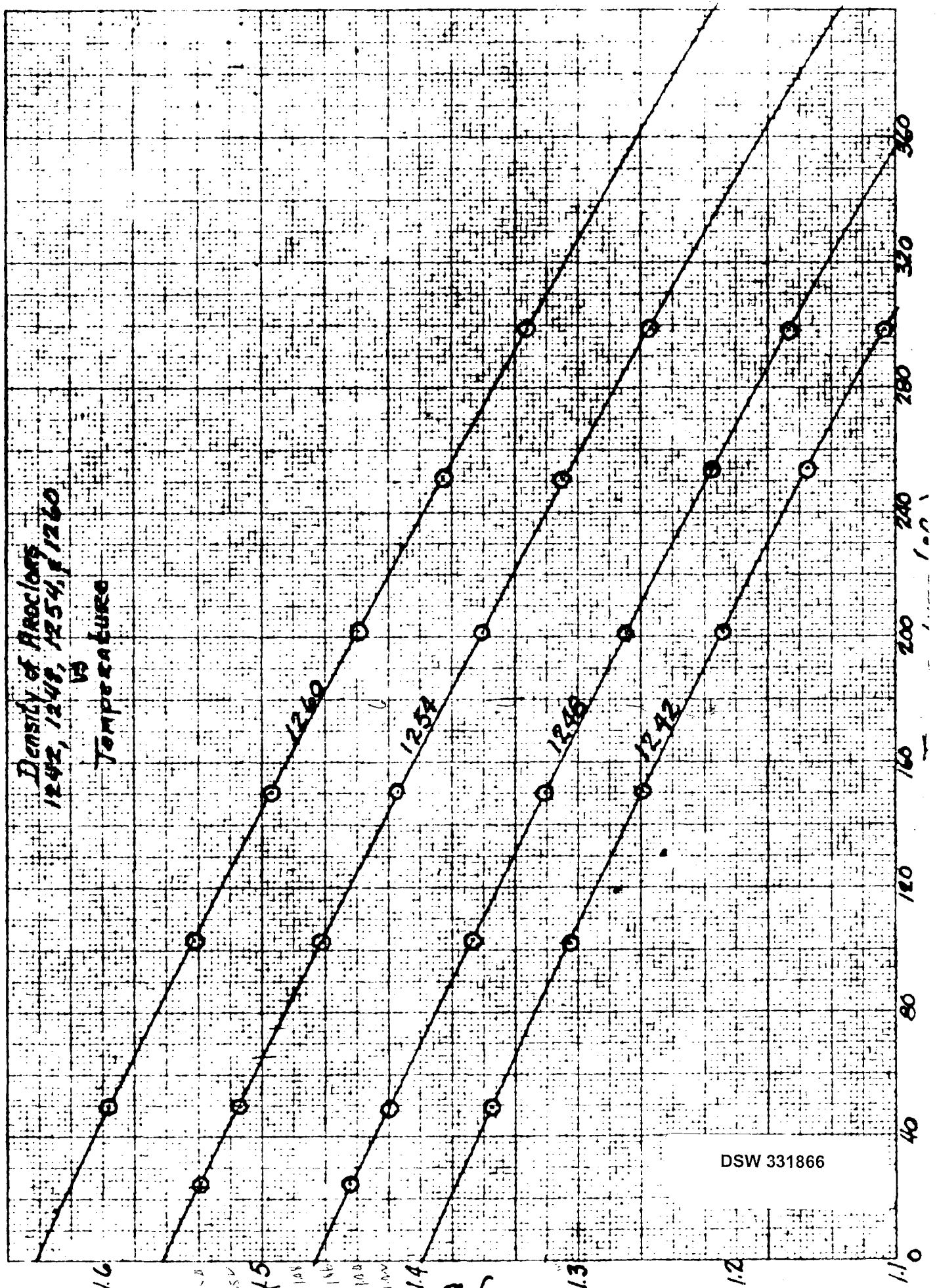
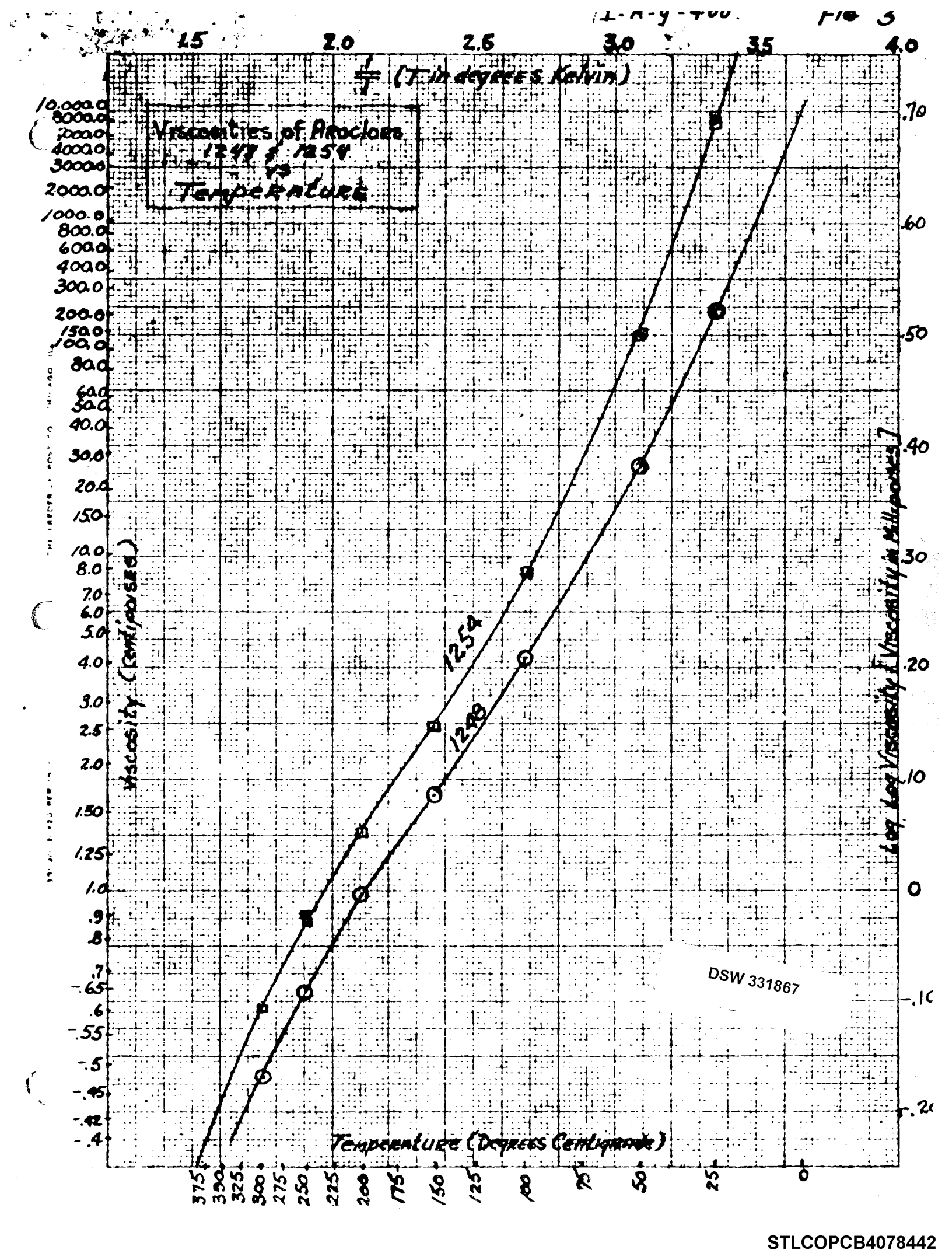


Fig. 1





DIRECTIONS FOR TEST NO. 14-32-48

SUBJECT: Evaporation Test of Liquid Aroclors

METHOD: 6 Hours Heating at 100° C. ASTM D6-39T - Modified

Procedure

Weigh on a rough balance about 50 grams (± .5 gm.) of the well mixed Aroclor into and accurately tared tin box, 55mm. dia. X 35 mm. deep (3 oz. Gill-style ointment box, deep pattern), Fisher #1-520. Let stand until box and sample are at room temperature, then weigh accurately.

Place the box in a ventilated convection oven maintained at 100° C ± 1° for 6 hours. Remove, cool in desiccator to room temperature and accurately reweigh. From the loss in weight calculate the evaporation in per cent. Report to second decimal place only.

Notes

1. Be sure box is at room temperature whenever exact weight is taken.

2. The oven should not contain other samples which might interfere with evaporation.

3. Because the evaporation loss is sensitive to temperature, the air bath must be closely maintained at 100° C.

FAB:cm
1-29-45

Copied by af
11/1/51

DSW 331868