ORGANIC

CHEMICALS DIVISION

RESEARCH

St. Louis Research Report No. 2420

FINAL REPORT

ON

ASSISTANCE TO SALES AND DEVELOPMENT DEPARTMENTS ON AROCLORS, BIPHENYL AND RELATED PRODUCTS - 1954

JOB NO. 2-02-750.01-2681

January 8, 1960

Written by: A.M. Ellenburg

Monsanto

St. Louis 66

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MONSANTO CHEMICAL COMPANY
ORGANIC CHEMICALS DIVISION
St. Louis Research Department

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INTRODUCTION

During 1954 many requests for physical properties and other data on the Aroclor series of compounds were received through our Sales Department. The information reported here has been used by Sales to assist in the marketing of Aroclors, biphenyl and related products.

SUMMARY

The requests by individual companies will be fully discussed in the experimental section of this report.

CONCLUSIONS

Any conclusions are expressed in the various projects written about in the report proper.

RECOMMENDATIONS

It is recommended that engineering assistance be obtained by the Sales Department to allow a more thorough exploitation of the use of Aroclor 1248 in heat transfer applications.

REFERENCES

St. Louis Research Report No. 1306 - Aroclor 1248 as a Heat Transfer Medium - August 26, 1955, A.M. Ellenburg, Job No. 2-02-750.01-2957.

EXPERIMENTAL WORK

A statement of the request and the work done to obtain the information will be reported fully for each company in separate sections of this part of the report.

A. Electrical Utilities Company, LaSalle, Illinois manufactures capacitors using Aroclor 1242, Aroclor 1254 and Pyranol 1481 (mixture of Aroclor 1254 and trichlorobenzene) as the impregnant. Coefficients of expansion for these products was requested over the temperature range of 25 to 70°C.

Using the pycnometer method, three density determinations were made at 25 and 70°C. for Pyranol 1481.

	Coefficient of Expansion Determinations		
Pyranol 1481	No. 1	No. 2	No. 3
Density at 25/25°C.	1.5188	1.5193	1.5185
Density at 70/70°C.	1.4697	1.4706	1.4703
Coefficient, cc/cc/°C.	0.000742	0.000736	0.000729

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The calculations were made using the formula, $d_{25} = d_{70} / [1 + \alpha (70-25)]$, where α is the coefficient of expansion.

The Aroclor booklet lists the coefficients for Aroclors 1242 and 1254 in the 25 to 65°C. range. Data sent to Electrical Utilities were as follows:

	Coefficient of Expansion	
	cc/cc/°C.	cc/cc/°F.
Aroclor 1242 (25 to 65°C.)	0.00068	0.00038
Aroclor 1254 (25 to 65°C.)	0.00066	0.000367
Pyranol 1481 (25 to 70°C.)	0.000735	0.000408

Phosphate Division NB No. 99761.

B. Samples of chlorinated biphenyl were obtained from Italian and French producers for comparison to our Aroclor 1254. The Italian samples were from the Societa Elettrica ed Elettrochimica del Caffaro of Milan and their sample is called Fenclor 54. The quality of this sample is much improved over previous samples evaluated in 1952. Prodelac of Paris, France produces Pyralene 1476 which is a high quality material. Small quantities of these samples were received, so only a limited analysis could be made for comparison.

Property	Aroclor 1254 Lot 1932	Fenclor 54	Pyralene 1476
Corrosion Test-210°C., 6 hrs.			
Loss of Aluminum	None	None	None
Acidity, mg. KOH/gm.	0.001	0.001	0.001
Inorganic Chlorides, ppm.	0.05	0.15	0.05
Color, APHA	50	50	30
Dielectric Constant 60 cycles, 100°C.	4.33	4.28	4.33
Power Factor 60 cycles, 100°C.	4.1%	4.5%	3.4%
Resistivity, 100°C. 500 volts DC, ohm-cm.	1200×10^9	1420×10^9	4800×10^9

The limited comparison shows that these two foreign producers are supplying chlorinated biphenyl to their customers that is equivalent to our Aroclor 1254. This situation, of course, affects our ability to sell Aroclor in these foreign markets.

Phosphate Division NB No. 99766-99767-99768.

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C. A Wagner Electric Company 7500 KVA transformer at Monsanto No. 4 substation in the J.F.Q. Plant was down for inspection. This unit stepped 66,000 volts down to 3300 volts. Samples were taken from the transformer and from the switch gear box for examination.

	Pyranol 1467	
	Transformer	Switch gear
Condition	Clear	Black
Resistivity, 100°C.	282×10^9	0.02×10^9
Power Factor, 1 kc, 100°C.	0.47%	>60%
Water, ppm.	25	20
Dielectric Strength, KV	45	29
Acidity, mg. KOH/gm.	--	0.16
Fe, ppm.	--	35

No doubt the iron in the switch gear Pyranol was the result of arcing and subsequent rusting of the steel. Mineral oil was used to replace the Pyranol in the switch gear box. The transformer sample was in excellent condition and required no treatment.

Phosphate Division NB No. 99778

D. Sangamo Electric Company, Marion, Illinois began studies on the use of Aroclor 1242 for capacitor impregnation. Difficulty was encountered in upgrading a drum of Aroclor 1242, Lot TC-3986. Samples were sent to St. Louis for tests.

After checking the resistivity of the material as received, 1000 grams of the Aroclor 1242 was stirred with 0.1% earth for 2-1/2 hours at 70-100°C. A second batch was treated similarly with 0.5% earth. The attapulgus earth was filtered out and the resistivity of the filtered Aroclor was checked.

Sample	Resistivity at 100°C.
Original Aroclor 1242	96×10^9 ohm-cm.
Original Aroclor 1242	165×10^9
Treated Aroclor 1242 (0.1% earth)	4100×10^9
Treated Aroclor 1242 (0.5% earth)	$18{,}800 \times 10^9$

Samples of our earth and our cell cleaning methods were sent to Sangamo for their use in solving the problem.

Phosphate Division NB No. 99784.

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E. Electrical Utilities Company of LaSalle, Illinois requested the vapor pressure of Pyranol 1481 in the temperature range of 0-50°C. Since no measurements had been made of the desired property, calculations were made from the vapor pressures of the components, 75% Aroclor 1254 and 25% trichlorobenzene by weight.

Vapor pressure for 1,2,4-trichlorobenzene was taken from Chemical Engineering, Vol. 61, April, 1954, p. 154.

The formula for the vapor pressure of Aroclor 1254 is:

$$\log P = -\frac{3780}{T(°K)} + 8.62$$

Pyranol 1481

Temperature	Partial Pressure TCB	Partial Pressure Aroclor 1254	Total Pressure Pyranol 1481
112°C.	13.12 mm.	.039 mm.	13.16 mm.
50	.76	.00051	.76
40	.34	.00022	.34
30	.23	.000086	.23
20	.114	.000032	.114
10	.057	.000011	.057
0	.027	.0000036	.027

Partial pressures were obtained by multiplying the vapor pressure of the components by the mole fraction present, 38% mole fraction TCB and 62% mole fraction Aroclor 1254.

It is evident that the trichlorobenzene contributes most of the vapor pressure of Pyranol 1481 in the temperature range of 0 to 50°C.

F. As a result of a flash fire in the exhaust scrubber of an air compressor using Aroclor 1254, Mr. R.B. Sykes of Pan-Am Southern Corporation Destrelan, Louisiana informed us that Aroclor 1254, Lot 105, had a flash point. The reserve sample of Lot 105 and a returned sample from Pan-Am both confirmed the presence of a flash point of 360°F. for our non-flammable Aroclor 1254.

Infrared studies, distillation and odor tests all confirmed the fact that about three percent biphenyl had contaminated this lot of Aroclor. From Anniston, it was learned that this Aroclor had been pumped into a storage tank which had previously contained biphenyl. Apparently all of the biphenyl had not been cleaned out completely.

As a step to prevent the passing of Aroclor by our laboratory, flash point determinations are now made on each lot of Aroclors 1242 and 1254 produced.

Phosphate Division NB NO. 99794.

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G. General Electric Company of Hudson Falls, New York has been using a mixture of Aroclor 5460 and Aroclor 1260 (1:2) as a potting compound. Up to now, G.E. has been making their own mixture and have rejected several lots of Aroclor 5460 because of solid matter which contains iron, non-magnetic dust and fibrous material. No doubt some of the contaminants come from the fiber drums used for packaging Aroclor 5460.

A practical solution to the problem is to mix the Aroclors at Anniston, then filter the mixture. A laboratory mix of this new Pyranol 1492 was made, heated to 200°C. with 0.1% attapulgus earth and filtered. The resistivity was increased from 25 x 10^9 ohm-cm. to a high value of 50,000 x 10^9 ohm-cm. at 100°C. by the filtering treatment. G.E. was sent a sample of the proposed mixture for acceptance tests.

Specifications were set and plant production of this Pyranol 1492 was begun.

Composition by weight	
Aroclor 5460	33.3%
Aroclor 1260	66.7%
Properties	
Color	Pale Yellow
Flow Point (Ball and Ring)	45-50°C.
Dielectric Strength	30 KV min.
Condition	Clear
Resistivity, 100°C.	500 x 10^9 ohm-cm., min.
Reference Test Methods	
Sampling	ASTM D901
Dielectric Strength	ASTM D924
Resistivity	G.E. Method E4D2-S1
Flow Point	ASTM E28

St. Louis Research NB No. A-57454.

H. Physical Constants of Aroclor 1248 were assembled and graphs were made of several properties over the range of 0 to 600°F. These data are specifically for the Aroclor 1248 - Heat Engineering Data Book, which Monsanto is publishing to assist in the design of Aroclor heaters for heat transfer medium.

These plots are attached to this report.

I. Allis-Chalmers Manufacturing Company of Milwaukee, Wisconsin submitted samples of Aroclor 1242 which they had extracted from Westinghouse and General Electric capacitors.

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The request was made for assistance in determining what produced the yellow color in the Westinghouse sample. Since it is known that Westinghouse uses anthraquinone in capacitor fluid, an ultraviolet absorption spectrum was used to detect the presence of about 0.2% anthraquinone in the Westinghouse sample. None was found in the General Electric material.

Dr. Keller reports that a simple test for anthraquinone was also devised. A small amount of sample is mixed in a test tube with an equal amount of concentrated H_2SO_4 by stirring vigorously with a stirring rod. A positive test at the 0.1 to 1% level is the formation of a bright yellow H_2SO_4 phase which when standing becomes orange at the interface. A negative test is indicated by a colorless H_2SO_4 phase. The sensitivity of the test method has not been established. The test is not necessarily specific for anthraquinone and it is conclusive only in the case of a negative test.

DISCUSSION

In addition to the laboratory work presented, several visits were made with Mr. P.G. Benignus to firms interested in the use of Aroclors for various applications.

Cleaver Brooks Company, Milwaukee, Wisconsin was visited in connection with the use of Aroclor 1248 in their heater for barges handling asphalt and bituminous materials.

International Boiler Works Company, East Strondsburg, Pennsylvania was visited in connection with the use of the Lamont forced circulation heater for Aroclor 1248 in heat transfer applications.

Electrical Utilities Company, LaSalle, Illinois; Gudeman Electric Company, Chicago, Illinois; General Electric Company, Hudson Falls, New York; Sangamo Electric Company, Marion, Illinois and Allis-Chalmers Manufacturing Company, Milwaukee, Wisconsin were visited in the interest of the use of Aroclor 1242 for capacitor manufacture. It was about this time that the entire industry was switching from Aroclor 1254 to the more fluid Aroclor 1242 for lower temperature use.

National Cash Register Company of Dayton, Ohio was getting into large scale production on their carbonless reproducing paper. A visit to their plant was made to discuss the handling of tankcar quantities of Aroclor 1242.

Several other firms sent representatives to St. Louis to discuss Aroclor applications mainly in the design of plants to use Aroclors for capacitor impregnation. Mr. Robert T. Sutherland of Starring and Company, Inc. (now Ventures, Inc.), Bridgeport, Connecticut discussed their plant for capacitors. Since 1954, the plant was built and is now in production of capacitors.

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Mr. Jack Devine and Mr. F. W. Robinson of Delco Products Division of General Motors Corporation, Dayton, Ohio made two visits to St. Louis to discuss Aroclor capacitor plant design. So far, their plans have not materialized to build a plant.

Mr. M. Meyer of the Wells Manufacturing Company, Foud du Lac, Wisconsin was interested in the use of Aroclor 1254 for ignition capacitors for Ford automobiles. Advice was requested several times during the startup of their operation, but it has been producing excellent capacitors.

The Research Group has been glad to cooperate with the Sales Department, especially Mr. P.G. Benignus in promoting a greater and more varied use of the Aroclors.

A. M. Ellenburg

A. M. Ellenburg

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APPENDIX

Graph 1 - Density of Aroclor 1248

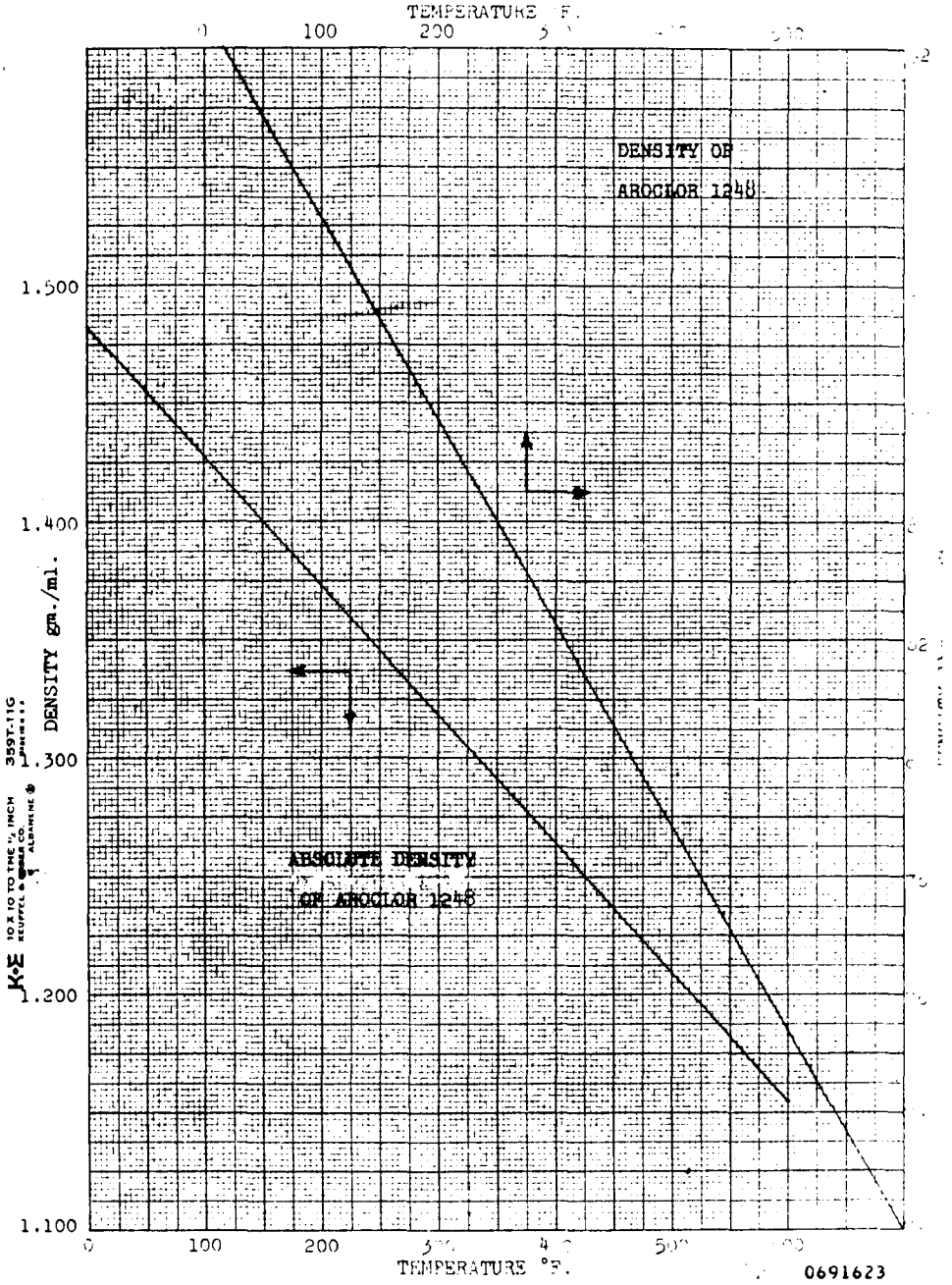
Graph 2 - Specific Heat of Aroclor 1248

Graph 3 - Comparison of Specific Heat of Thermal Fluids

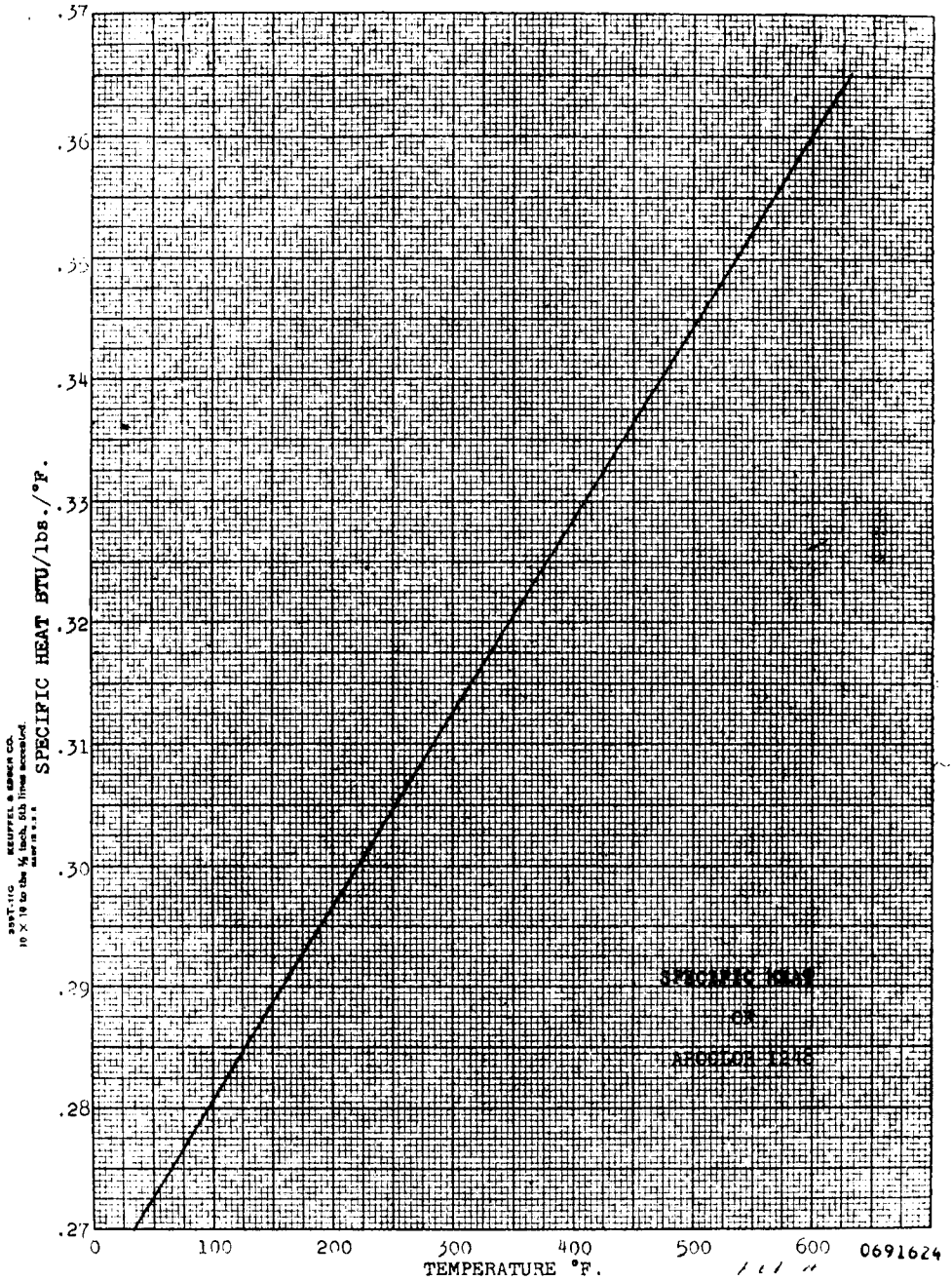
Graph 4 - Viscosity of Aroclor 1248, absolute

Graph 5 - Viscosity of Aroclor 1248, Saybolt Universal Seconds

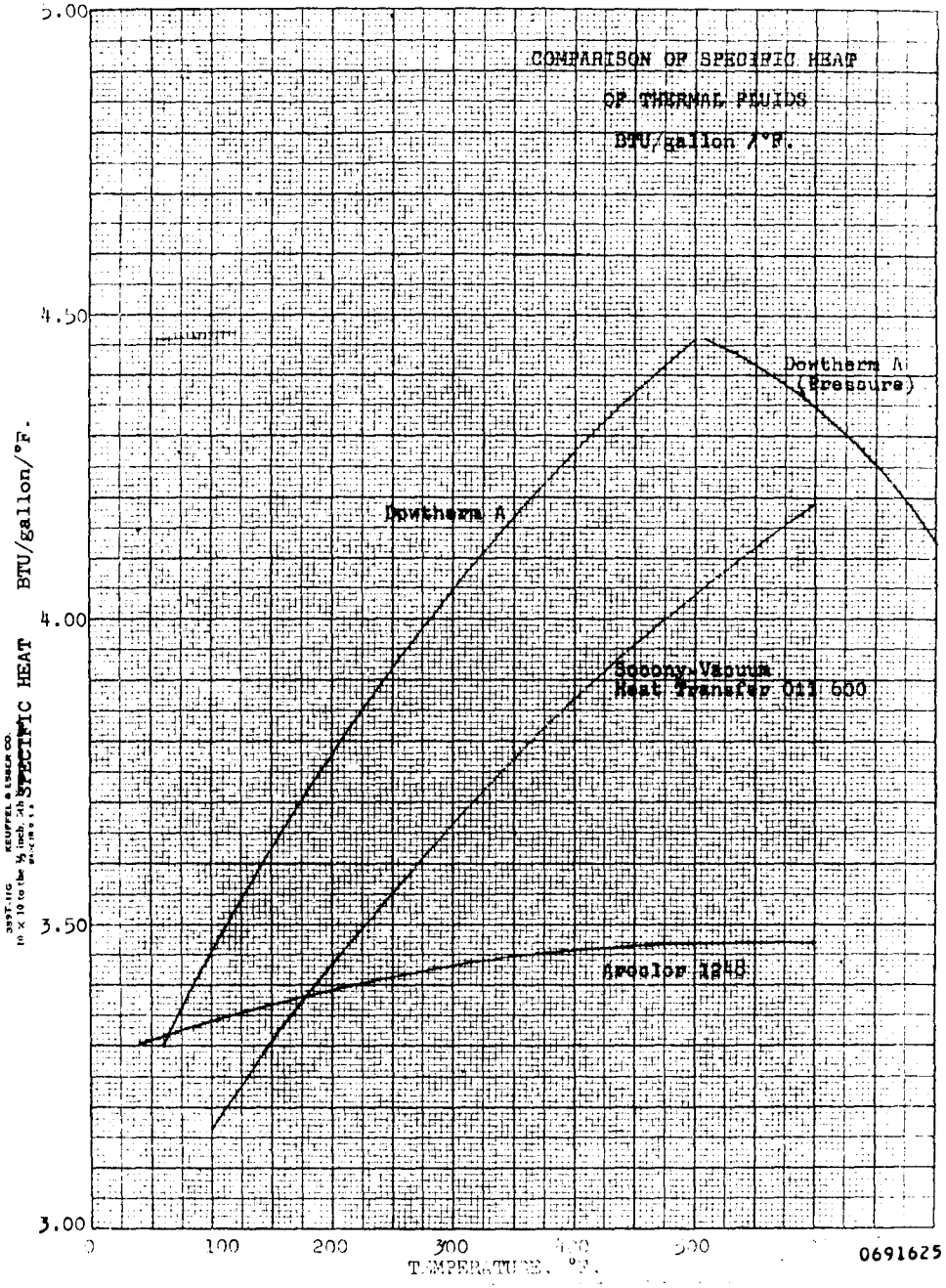
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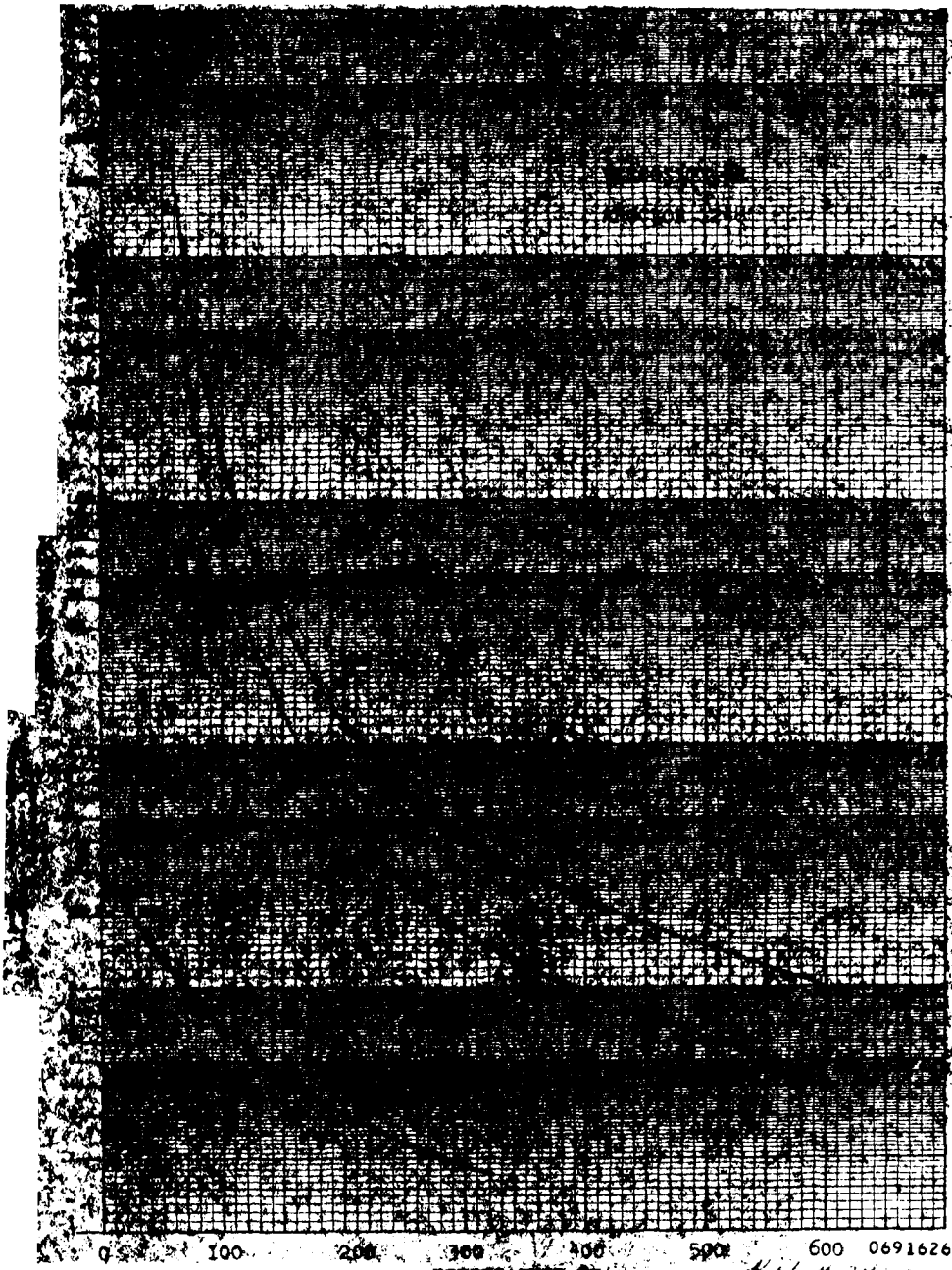
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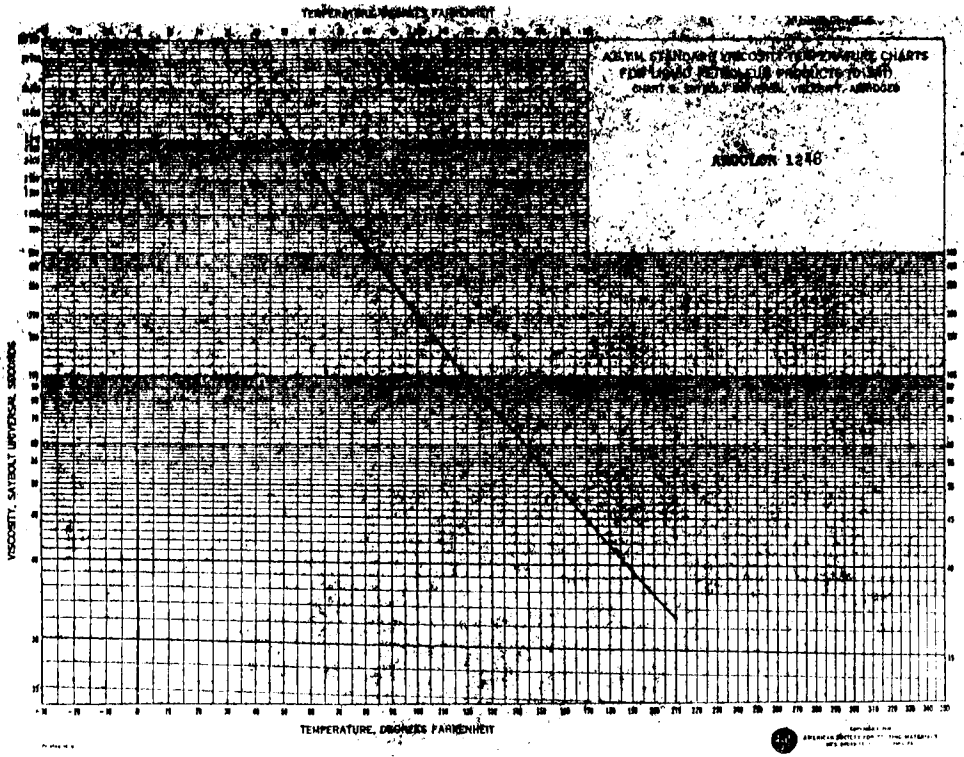
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