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NAVY DEPARTMENT

Report on

The Effect of Chlorinated Saturants on the Mechanical and Electrical Properties of Polyvinyl Chloride Type Insulation

NAVAL RESEARCH LABORATORY
ANACOSTIA STATION
WASHINGTON, D. C.

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ABSTRACT

The production of some types of Navy electrical cables was stopped temporarily because apparently there was penetration of the primary synthetic resin insulation around the conductors by the saturant used in the asbestos rovings and fillers of the cable. To help clarify this situation tests were made on the rate at which a chlorinated cable saturant, Halowax No. 2084, would penetrate a synthetic resin insulation of the type used in these cables, Vinylite 5901. (Vinylite 5901 consists principally of tricresyl phosphate and copolymerized vinyl chloride and vinyl acetate). The results indicate that the Halowax will not penetrate the Vinylite appreciably at room temperature but will penetrate it slowly at 50° C and rapidly at 100° C.

The effects of the Halowax on the mechanical and electrical properties of the Vinylite were also observed. The Halowax improved the electrical properties of the Vinylite in some respects but it injured the insulation mechanically in that it made the insulation stiffer and increased the temperature at which it cracked on bending. This decrease in flexibility was the only damaging effect noted.

INTRODUCTION

(a) Authorization

1. This work was done in conjunction with the development methods of test for electrical insulation authorized by Bureau of Ship's letters S62(11)(350) dated November 16, and November 24, 1942, to Naval Research Laboratory. These letters cover specific directives Nos. 12, 13, and 14.

(b) Statement of Problem

2. The purpose of this work was to determine the rate at which a chlorinated saturant (Halowax #2084) will penetrate an adjacent layer of polyvinyl chloride insulation (Vinylite 5901) and the extent to which the properties of the insulation will be altered by the saturant.

METHODS

(a) Materials

3. SRI - 3(7) unit conductor manufactured by the Anaconda Wire and Cable Company. The insulating coating was black Vinylite #5901. Part of the insulated conductor was wrapped with asbestos and saturated with Halowax #2084 at the factory.

(b) Procedure

4. The work on this wire may be separated into two parts. The first part concerns the extent and conditions under which Halowax diffuses into Vinylite insulation. The second part concerns the effect of given amounts of Halowax on the mechanical and electrical properties of the insulation.

5. The work in the first part involved observing the gain in weight of Vinylite when it was left in contact with Halowax at various temperatures for various times. The work in the second part involved making the following mechanical and electrical tests:

(a) Tensile strength at room temperature.
(b) Elongation and tensile strength at 80°C.
(c) Brittle point tests at low temperatures.

(d) Dissipation factor and insulation resistance measurements in the temperature range of 15 to 120°C.

The detailed procedures for all these tests are given in the appendix under their respective headings.

DATA OBTAINED

(a) Curves or plots of data.

6. Plate 1 shows curves of gain in weight versus time for Vinylite when in contact with Halowax at various temperatures. Each point on the curves represents a separate sample. No samples were returned to the oven after they were removed for weighing but they were set aside to be used in the tensile, elongation, and brittle point tests.

7. Plate 2 shows curves of per cent elongation versus time for a number of Vinylite wire coverings when stretched under constant load at 80°C. Some of the samples used were those saved from the gain in weight measurements. They contained various amounts of Halowax.

8. Plates 3 and 4 give curves showing the effect of Halowax on the dissipation factor of Vinylite over a temperature range of 100°C at 60 cycles and at 10,000 cycles, respectively. The two nearly identical curves having the highest dissipation factor peaks on each plate represent two samples treated identically in order to test the reproducibility of the method.

9. The curves on Plate 5 show the effect of Halowax on the insulation resistance of Vinylite. The same samples were used here that were used for the dissipation factor measurements.

(b) Data in Tabular Form.

10. Table I contains data on the gain in weight of Vinylite insulation when wrapped with Halowax impregnated asbestos and heated at various temperatures. Table II contains data on the chlorine content of plasticizer extracted from the Vinylite insulation referred to in Table I. Table III contains the results of calculations, using the data in Tables I and II, to give the amount of exchange of Halowax and plasticizer between the

the asbestos and the Vinylite layers on the wire. Details of the calculations and discussion of results are given in Part I of the appendix.

11. Tables IV, V, and VI give the results of mechanical tests on Vinylite insulation containing various amounts of Halowax. Table IV covers tensile strength at room temperature, Table V covers elongation and breaking time under constant load at 80°C, and Table VI covers brittleness tests at low temperatures. See Part II A, B, and C of the appendix for details.

CONCLUSIONS AND RECOMMENDATIONS

(a) Facts Established

12. Halowax 2084 diffuses into Vinylite 5901 increasing rapidly as the temperature is raised above 50°C. There is practically no transfer at room temperature.

13. Vinylite insulation is made stiffer by the presence in it of 15% Halowax. This somewhat increases the tensile strength at room temperature but has little effect at 80°C. The most pronounced effect of the Halowax is that it increases the temperature of the brittle point. The brittle point of untreated Vinylite was -24.5°C while that containing around 15% Halowax was -13°C.

14. Halowax shifts the peak in the dissipation factor temperature curve of Vinylite upward and to the right (higher temperature). It also shifts the insulation resistance-temperature curve up. The insulation resistance of Vinylite 5901 with 15 per cent Halowax #2084 in it is about 10 times that of untreated Vinylite.

(b) Opinions and recommendations

15. The only definitely deleterious effect of the Halowax on Vinylite was the decrease in low temperature flexibility. It is recommended, therefore, that the use of cables containing Halowax in direct contact with polyvinyl chloride type insulation be avoided in locations where they will be subjected to bending at temperatures below -10°C(14°F).

SUMMARY

16. The rate of intrusion of Vinylite 5901 by an adjacent layer of Halowax was found to be negligible at room temperature. At 50°C the rate of penetration was still comparatively slow but it increased rapidly in going to higher temperatures.

17. The over-all electrical properties of Vinylite are not seriously impaired by Halowax. In fact, Halowax produces a distinct improvement in insulation resistance. Mechanically, Halowax causes Vinylite to become stiffer and the brittle point or cold crack temperature is raised. This decrease in flexibility was the only serious impairment noted. It was concluded that no difficulties would arise in bending the wire unless temperatures were below -10°C(14°F).

TABLE I

Gain in weight (%) of Vinylite Wire Coating
When Wrapped With Halowax Impregnated Asbestos

Time	25° C	50° C	70° C	100° C	135° C
15 hours					32.8
16-1/2 hours			2.1		
17-1/2 hours				10.4	
40 hours		.36	3.3	12.8	
57 hours				15.2	
73 hours				17.3	
92 hours		.50			
113 hours			4.2		
192 hours	.17				

TABLE II

Chlorine content of plasticizer extracted from Vinylite insulation given various Treatments.

	Chlorine mg/gm Vinylite layer	Chlorine % orig. weight of Vinylite
Control uncovered wire heated 39-1/2 hrs at 100° C.	8.4	0.84
Covered with Halowax impregnated asbestos at factory - Not heated.	22.3	2.23
Covered with Halowax impregnated asbestos - Heated 17-1/2 hrs. at 100°C - 10.4% gain in weight.	84.2	9.3
Covered with Halowax impregnated asbestos - Heated 39-1/2 hrs. at 100°C. 12.8% gain in weight.	103.0	11.6

TABLE III

Exchange of Halowax and Plasticizer Between Asbestos and Vinylite Coating.

Sample	Chlorine % orig. wt. of Vinylite	Halowax transferred to Vinylite - %	Plasticizer transferred to asbestos %
Heated 17-1/2 hrs. at 100° C. 10.4% gain in weight.	9.3	14.1	3.7
Heated 39-1/2 hrs. at 100° C. 12.8% gain in weight	11.6	17.9	5.1

- -

TABLE IV

Effect of Halowax on Tensile Strength of Vinylite 5901 at Room Temperature

Insulation Treatment	Tensile Strength - lbs.
No Halowax, no heating	15.5
No Halowax, heated 6 hrs. at 120° C plus 16 hrs. at 105° C	13.0
Halowax covered, no heating	15.8
Halowax covered, heated 72 hrs. at 90° C 10.5% increase in weight	17.0
Halowax covered, heated 6 hrs at 120° C plus 16 hrs. at 105° C 13.6% increase in weight	18.0

TABLE V

Elongation and Breaking Time of Vinylite Wire Coating
By 1,000 Gram Weight at 80° C.

Sample	Elongation in 5 min. - %	Elongation at break - %	Time of break minutes
Control	163 150	185 175	10.3 10.3
Control - heated 18 hrs. at 100° C.	107 117	145 131	21.4 9.0
Control - heated 43 hrs. at 100° C.	110 120	130 133	11.2 7.6
Wrapped - heated 64 hrs. at 70° C 3% gain in weight	116	163	18.3
Wrapped - heated 113 hrs. at 70° C 4.2% gain in weight	100	157	29.3
Wrapped - heated 57 hrs. at 100° C 15.2% gain in weight	116	129	9.8
Wrapped - heated 73 hrs. at 100° C 17.3% gain in weight	114	143	12.8

TABLE VI

Effect of Halowax on Brittleness of Vinylite 5901
at Low Temperatures

Insulation treatment	Brittle Point °C
No Halowax, no heating.	-24.5
Halowax covered (factory), no heating.	-23.0
Halowax covered, heated 88 hrs. at 50° C, 0.4% increase in weight.	-18.0
Halowax covered, heated 17-1/2 hrs. at 100° C, 10.4% increase in weight.	-13.0
Halowax covered, heated 39-1/2 hrs. at 100° C, 12.8% increase in weight.	-14.0
Halowax covered, heated 15 hrs. at 135°C. 33% increase in weight.	-11.0

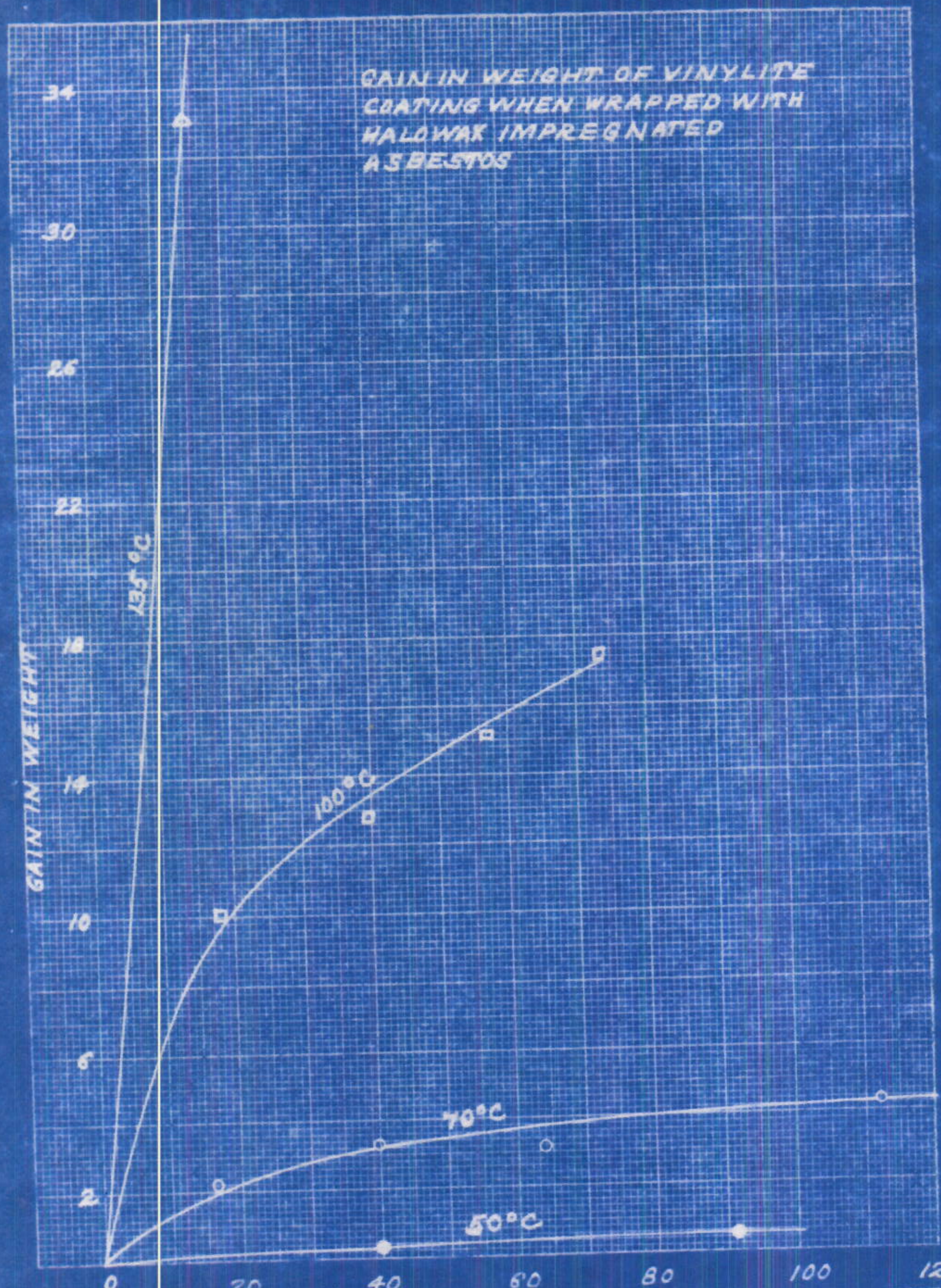
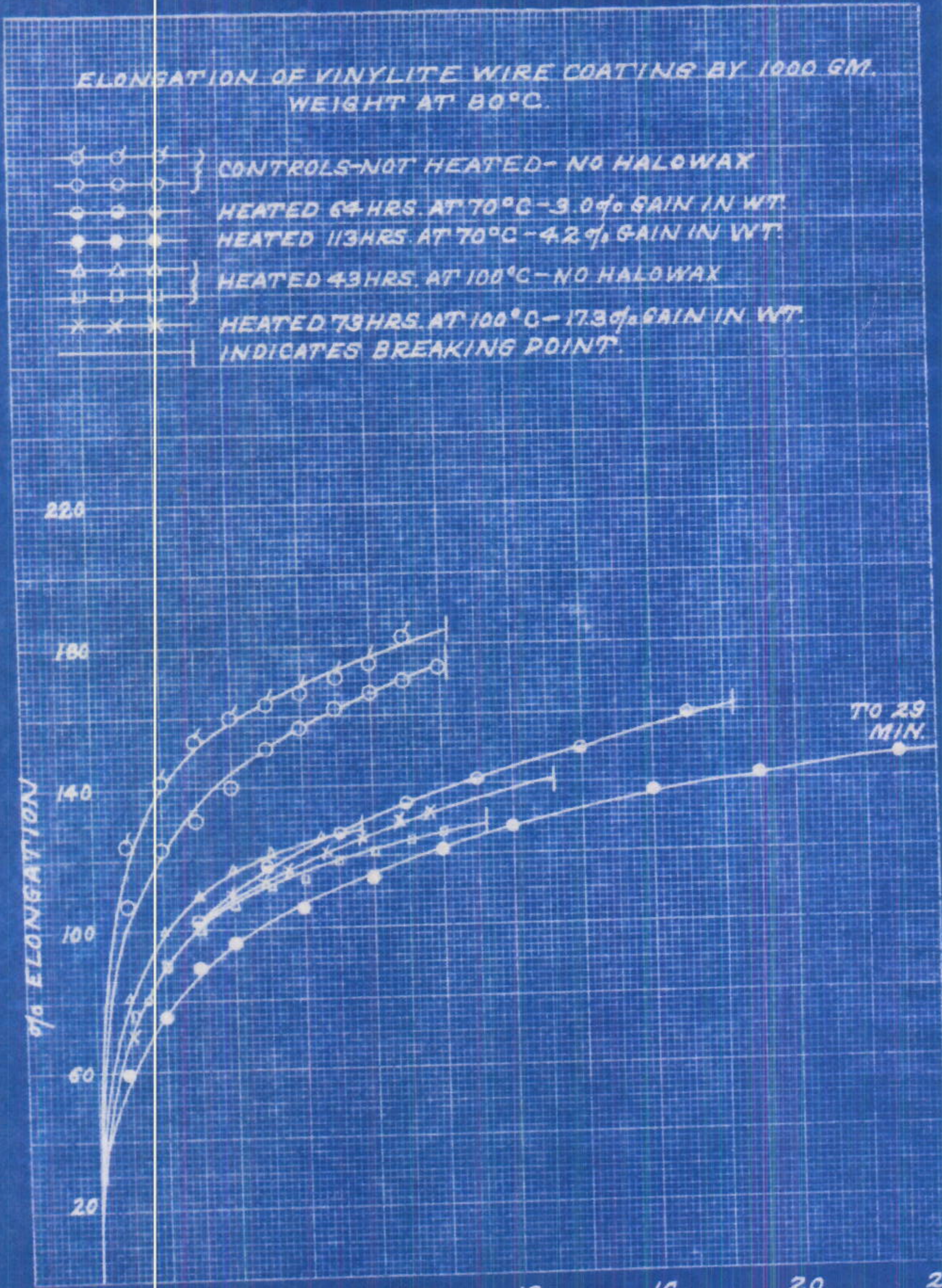
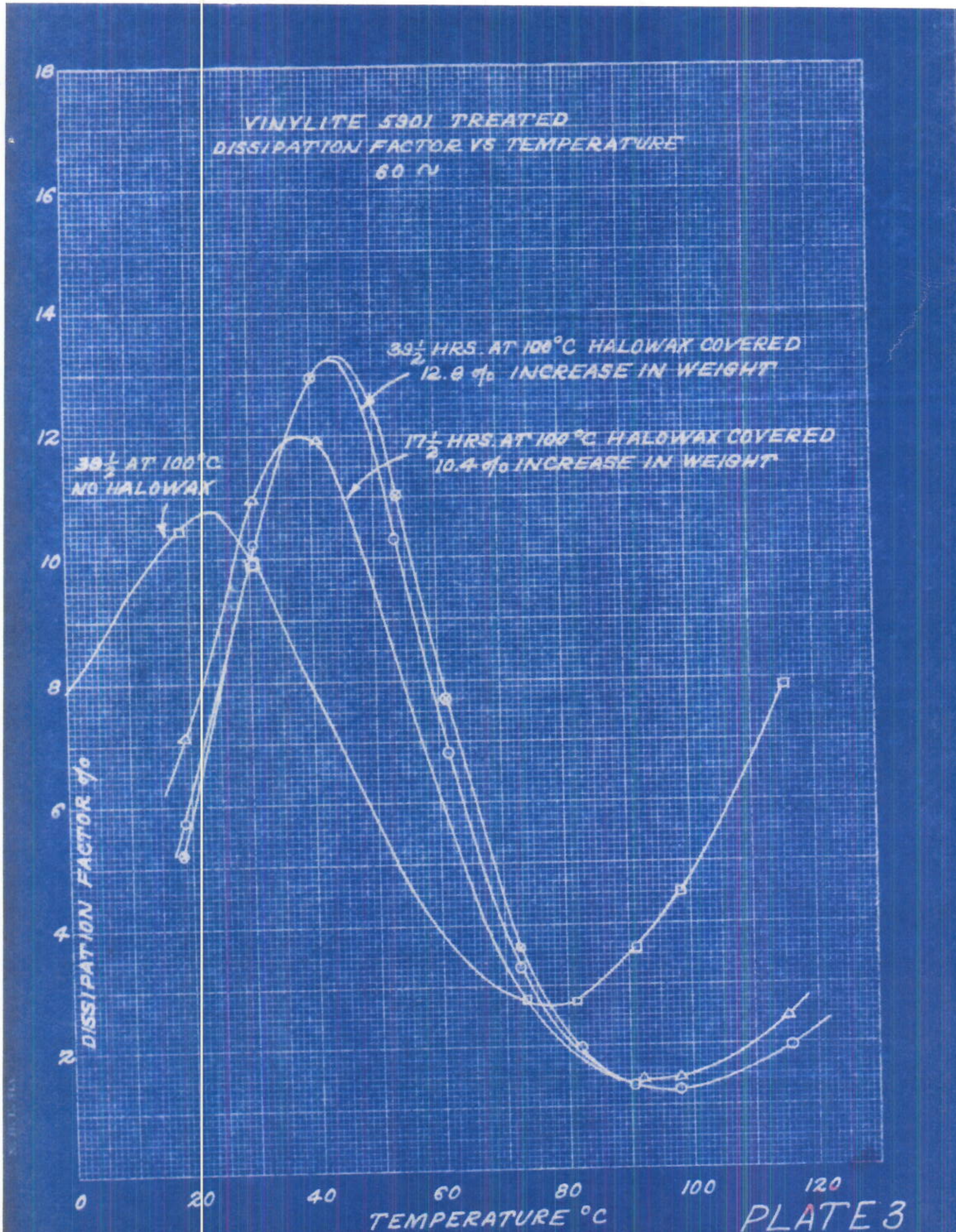

GAIN IN WEIGHT OF VINYLITE
COATING WHEN WRAPPED WITH
HALOWAX IMPREGNATED
ASBESTOS
34
30
26
22
18
14
10
6
2
GAIN IN WEIGHT
135°C
100°C
70°C
50°C
0
20
40
60
80
100
120
TIME IN HOURS

PLATE I



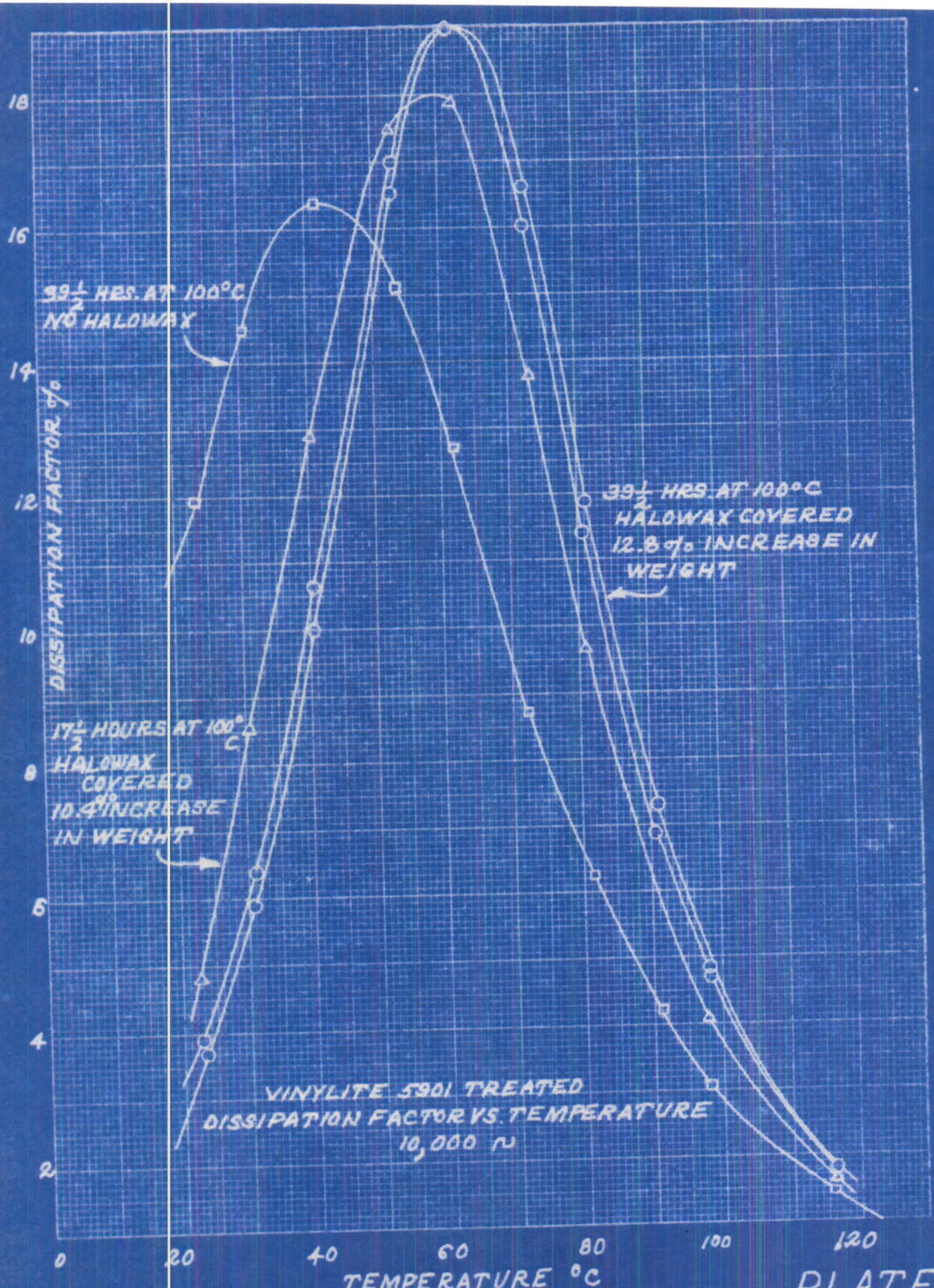
ELONGATION OF VINYLITE WIRE COATING BY 1000 GM. WEIGHT AT 80°C.
CONTROLS-NOT HEATED- NO HALOWAX
HEATED 64 HRS. AT 70°C - 3.0% GAIN IN WT.
HEATED 113 HRS. AT 70°C - 4.2% GAIN IN WT.
HEATED 43 HRS. AT 100°C - NO HALOWAX
HEATED 73 HRS. AT 100°C - 17.3% GAIN IN WT.
INDICATES BREAKING POINT.
TO 29 MIN.
% ELONGATION
220
180
140
100
60
20
0
4
8
12
16
20
24
TIME IN MINUTES

PLATE 2



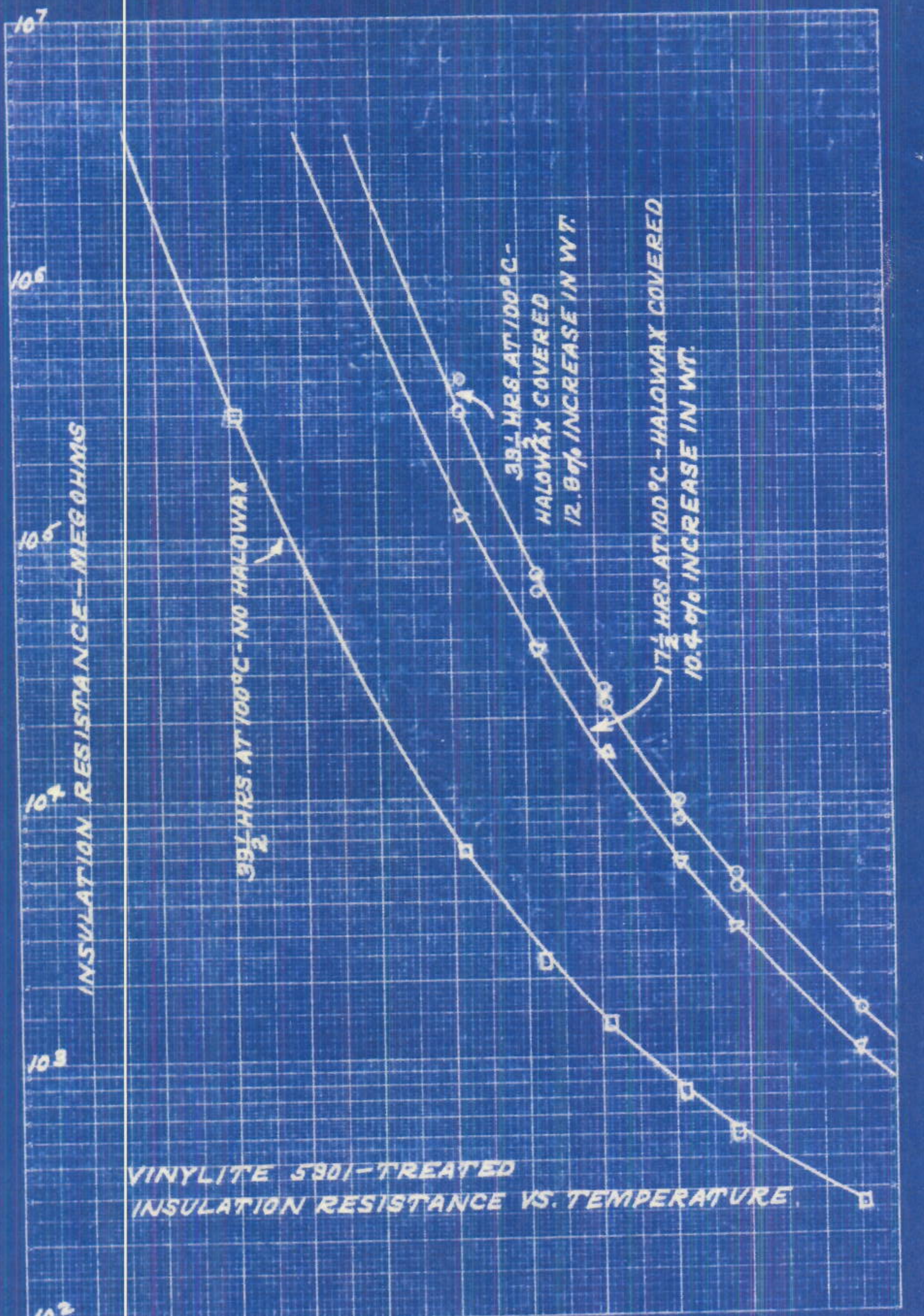
VINYLITE 5901 TREATED
DISSIPATION FACTOR VS TEMPERATURE
60 ~
39½ HRS. AT 100°C HALOWAX COVERED
12.8 % INCREASE IN WEIGHT
17½ HRS. AT 100°C HALOWAX COVERED
10.4 % INCREASE IN WEIGHT
39½ AT 100°C
NO HALOWAX
DISSIPATION FACTOR %
TEMPERATURE °C
0
20
40
60
80
100
120
2
4
6
8
10
12
14
16
18

PLATE 3



18
16
14
12
10
8
6
4
2
DISSIPATION FACTOR %
0
20
40
60
80
100
120
TEMPERATURE °C
39½ HRS. AT 100°C
NO HALOWAX
39½ HRS. AT 100°C
HALOWAX COVERED
12.8% INCREASE IN
WEIGHT
17½ HOURS AT 100°C
HALOWAX
COVERED
10.4% INCREASE
IN WEIGHT
VINYLITE 5901 TREATED
DISSIPATION FACTOR VS. TEMPERATURE
10,000 ~

PLATE 4



10^7
10^6
10^5
10^4
10^3
10^2
INSULATION RESISTANCE—MEGOHMS
39½ HRS. AT 100°C - NO HALOWAX
39½ HRS AT 100°C - HALOWAX COVERED 12.8% INCREASE IN WT.
17½ HRS AT 100°C - HALOWAX COVERED 10.4% INCREASE IN WT.
VINYLITE 5901-TREATED
INSULATION RESISTANCE VS. TEMPERATURE
20
40
60
80
100
120
TEMPERATURE °C

PLATE 5

APPENDIX

PART I

RATE OF TRANSFER TESTS

A. Penetration of Vinylite Wire Coating by Halowax

1. The purpose of these tests was to determine the extent of the transfer of Halowax from the asbestos roving to which it was originally applied as an impregnant to the Vinylite layer immediately under it. The effect of temperature on the rate of transfer was also studied.

a. Procedure

2. The degree of penetration of the Vinylite by Halowax was determined by direct weighing of the Vinylite-coated wire samples before and after periods of contact with Halowax-impregnated asbestos. Samples of Vinylite-coated wire 10 centimeters long and weighing approximately 2 grams were used for the test. Halowax-impregnated asbestos samples of the same length were carefully removed from portions of similar wire which had been covered by the cable manufacturer. The asbestos layer weighed approximately 1 gram per 10 centimeters length and contained about 0.4 gram of Halowax #2084.

3. After the Vinylite-coated wire samples had been cleaned by wiping with benzene and weighed, they were inserted into the asbestos roving which had been previously removed from other wire. Care was taken to insure complete covering of the Vinylite coating by the asbestos and the asbestos then bound spirally with thread in order to keep it in close contact with the Vinylite. The thread served the same purpose as the rayon braid used over the asbestos by the cable manufacturer. The approximate make-up of the samples thus prepared was as follows:

		Weight per 10 cm length
Impregnated asbestos	(Asbestos	0.6 gm
	(Halowax	0.4 gm
Vinylite		0.7 gm
7-strand wire		1.3 gm

4. The wrapped samples were placed in small glass tubes tightly stoppered to eliminate the possibility of loss in weight through volatilization at the higher temperatures. The rate of take-up of Halowax by the Vinylite was studied at 25°, 50°, 70°, 100°, and 135° C. The gain in weight of the Vinylite-coated wire was determined by removing the asbestos, wiping with benzene to remove adhering Halowax and weighing the sample. The gain was calculated as per cent of the original weight of the Vinylite layer - approximately 0.7 gram.

5. The method of direct weighing gives minimum values of degree of penetration of the Halowax since any migration of the plasticizer from the Vinylite coating is not detected as such. Chlorine determinations on the plasticizer fraction of the Vinylite coating, when considered in conjunction with the direct weighing results, permit the calculation of the exchange of Halowax and of plasticizer between the impregnated asbestos and the Vinylite coating. Several chlorine determinations were made by Mr. Clark of the Food and Drug Administration of the U. S. Department of Agriculture by a modified Parr bomb method. The determinations were made on plasticizer fractions obtained from the Vinylite coating by a method of selective solvents. This method is described in Naval Research Laboratory letter S62-2(9)(460) of April 21, 1943, to the Bureau of Ships.

b. Results

6. The results of the gain in weight determinations are given in Table I and are shown graphically in Plate 1. The results of the chlorine determinations are given in Table II.

7. Assuming that the difference between the chlorine content of the control and the samples heated in contact with Halowax-impregnated asbestos is due to the transfer of Halowax, the amount of transfer can be calculated. The chlorine content of Halowax #2084 which was used as the impregnant for the asbestos is calculated to be approximately 60%. This checks with the results of chemical analysis of the material extracted from the asbestos.

8. The percentage of Halowax transferred, as calculated from chlorine determinations, is higher than can be accounted for by the actual gain in weight of the Vinylite layer. The most plausible explanation of this discrepancy is that there is also a transfer of plasticizer from the Vinylite to the impregnated asbestos. The results of the calculations of the exchange of plasticizer and

Halowax for two samples at 100° C are shown in Table III

c. Discussion of Results

9. The increase in weight of the Vinylite wire coating shows that transfer of Halowax from the asbestos to the Vinylite takes place in amounts which are readily detectable. The rate of transfer varies considerably with temperature. At temperatures of 50° C and below where the Halowax in question is extremely viscous, the rate is slow. The rate is much more rapid at temperatures of 70° C and above, at which temperatures the Halowax becomes quite fluid. The gain in weight of the Vinylite layer in extreme cases represents more than half of the Halowax originally present in the asbestos roving.

10. The calculation of the exchange of Halowax and plasticizer from a consideration of gain in weight data and chlorine determinations is a valid procedure providing that the Halowax going into the Vinylite is of the same chlorine content as that originally present in the asbestos. The Halowax #2084 reported by Union Carbide and Carbon Corporation to be used in this case is made up of chlorinated naphthalene 62 - 64% Cl, chlorinated diphenyl 60% Cl and polystyrene. The latter ingredient contains no chlorine but is present only to the extent of 5 per cent. It is reasonably safe to assume, therefore, that the Halowax migrating into the Vinylite layer is very nearly of the same chlorine content (60%) as that originally present in the asbestos. On the basis of the chlorine analysis and the gain in weight results for several samples heated at 100° C, it was calculated that the weight of plasticizer leaving the Vinylite layer is approximately 1/4 to 1/3 of the weight of Halowax entering it. This relationship would not necessarily be true at other temperatures.

11. No gain in weight data were obtainable to determine the amount of Halowax in the Vinylite layer of the wire that had been covered with impregnated asbestos at the factory for the reason that the original weight before covering was unknown. Chlorine analysis of the plasticizer fraction as shown in Table II indicates the presence of approximately 3.7% Halowax in the factory-covered sample. The chlorine in this case was calculated as coming entirely from the Halowax, since the sample was not heated except during the application of the impregnant to the asbestos. According to information received from the manufacturer the Halowax is applied in toluene solution and the solvent evaporated at 80° C.

12. When the factory-covered Vinylite-coated wire was stripped of its impregnated asbestos and cleaned, weighed, and covered again with Halowax-impregnated asbestos, it was found that the gain in weight was slower than with the wire which had not been previously covered. This would seem to indicate that some Halowax was already in the Vinylite layer. This is in agreement with the results of the chlorine determination.

PART II

MECHANICAL AND ELECTRICAL TESTS

A. Tensile Strength Tests at Room Temperature.

Procedure

13. The tensile measurements were made on Vinylite insulation which had been treated in various ways as described under the gain in weight determinations in Part I. The measurements were made at room temperature on a Scott tester, Model U, of 50 pounds capacity and pulling speed of 20 inches per minute.

14. The samples were prepared for test by slipping the stranded conductor out of its covering, leaving the insulation undamaged. The tubes of insulation were then fastened in the tester, one at a time, and pulled apart. No breaks occurred at the clamps.

Results

15. The results, given in Table IV, show the tensile strength of Vinylite to be increased by the presence of Halowax.

B. Elongation and Tensile Strength Tests at 80° C.

16. Although the presence of Halowax in the Vinylite coating was not found to cause a lowering of the tensile strength at room temperature, there was a possibility that it might produce a different effect at temperatures above the melting point of the Halowax preparation used. A temperature of 80° C was chosen for the measurements; a temperature at which Halowax #2084 is very fluid. Tensile strength measurements were not made in the usual manner because of certain difficulties involved, and also because the extent of elongation at 80° C was sufficient to make actual tensile strength data obtained in the usual way of doubtful significance.

Procedure

17. Samples of Vinylite-coated wire which had previously been used for gain in weight determinations were used for these measurements. The stranded wire was pulled out from each sample without damaging the Vinylite coating,

leaving a tubular sample 10 centimeters in length. Screw clamps were attached to each end of the sample so as to leave an effective length of 7 centimeters for elongation and breaking time measurements. A 1,000 gram weight was attached to one clamp; a supporting wire, and a millimeter scale was attached to the other. The sample was placed in an oven having a glass door and operating at 80° C. After an interval of seven minutes, to permit the sample to reach the temperature of the oven, the supporting wire was pulled up through a small opening at the top of the oven until the pull of the 1,000 gram weight acted on the sample. The elongation of the sample was followed by observing the length on the millimeter scale which was suspended from the upper clamp along the length of the sample. The time at which the sample broke was also observed.

Results

18. Plate 2 shows the per cent elongation plotted against time. Curves are shown for controls which have been neither covered nor heated and also for controls which have not been covered, but have been heated at 100° C. Curves are also shown for samples which have been heated at 70° C and 100° C in contact with impregnated asbestos. The percentage gain in weight values is shown for these samples. The end of the curve indicates the breaking point of the sample.

19. Tabulated results are given in Table V. The elongation in five minutes, the elongation at the breakin point, and the time taken to reach the breaking point are shown.

Discussion

20. The results show that the sample which had not been heated in contact with Halowax-impregnated asbestos did not elongate as much as the unheated controls, but the difference in stretch cannot be considered to be caused by the Halowax. Control samples which were heated at 100°C also showed a decrease in elongation and no significant difference could be noted between the heated samples without Halowax and the heated samples which have gained as much as 17.3% in weight. The results indicate that the change in stiffness as observed at 80° C is more the result of prolonged heating than the result of the presence of Halowax.

21. The data on the time of breaking are not considered complete enough nor of sufficient reproducibility to be of much significance.

C. Brittle Point Determinations.

22. The brittle point tests were made on the same series of samples as were used for the tensile strength and elongation tests.

Procedure

23. A 10 centimeter length of Vinylite-covered wire was fitted inside a 12 centimeter length of small rubber tubing of 1/8-inch inside diameter and 1/16-inch wall thickness. The two ends of the tube were then closed with pinch cocks so the Vinylite was sealed inside. The tube was next placed in an alcohol - dry ice bath and brought to the desired temperature for testing. The tube was then removed quickly and bent at a rapid rate (180° in 1/5-second) around a 3/8" mandrel. The temperature is taken as being below the brittle point if the insulation cracks under those conditions.

24. The rate of bending of the tube around the mandrel is not critical as long as this rate is high. The brittle point obtained by this method is reproducible to within 1° C.

Results

25. The data given in Table VI show that the presence of Halowax in Vinylite has a marked effect on its brittle point. For example, the specimen which gained 10.4% (from imbibed Halowax) gave a brittle point 11.5° C higher than that given by the straight Vinylite.

D. Dissipation Factor vs. Temperature.

Introduction

26. The dissipation factor of polyvinyl chloride type insulation is very sensitive to the temperature and the frequency used in its measurement. When a plot is made of dissipation factor vs temperature at the proper frequency the dissipation factor will be found to go through a characteristic peak. The height and position of this peak is very dependent on the amount and type of plasticizer used with the resin. The presence of any Halowax in the Vinylite should show up, therefore, as a shift in the dissipation factor peak.

Procedure

27. Sprayed tin electrodes were applied to the outside of a 12-inch length of the Vinylite-covered wire using

a metal spray gun as described in Naval Research Laboratory letter S62-2(9)(460) of April 10, 1943, to the Bureau of Ships. The sprayed tin was scraped off the wire for about 1/2-inch back from each end and the specimen placed in an air thermostat. By means of guarded leads, a power factor bridge (General Radio Type 740-BG for 60 cycles and 716-A for 10,000 cycles per second) was hooked on to the specimen. The outer tin electrode was the grounded side, while the conductor inside the Vinylite was the high side. The temperature was varied over the range of 20° to 120° C and dissipation factor measurements made at various points along the way, both to 60 cycles and at 10,000 cycles per second.

Results

28. The data are given on Plates 3 and 4. The peaks occur at higher temperatures and higher dissipation factors at the higher frequency as compared to the lower frequency. The relative positions of the curves at a single frequency, however, appear practically the same at 60 cycles as at 10,000 cycles.

29. The presence of Halowax in Vinylite insulation shifts the peak in the dissipation factor-temperature curve up and to the right. This is shown by Plates 3 and 4. In order to show the reproducibility of the data, two identical samples of the wire which showed the 12.8 per cent increase in weight were measured. Both curves are given. There appears to be a rough proportionality between the gain in weight (amount of Halowax) in a given sample and the shift in the temperature at which the peak occurs in the dissipation factor curve.

E. Insulation Resistance

30. Insulation resistance measurements were made on the same specimens as were used for the dissipation factor measurements. The measurements were made with a General Radio Megohm Bridge, Type 544, using a potential of 100 volts. Data are given on Plate 5. It is seen that the insulation resistance of Vinylite 5901 is increased markedly by the addition of Halowax. There appears to be an approximate proportionality between the gain in weight of the sample and the increase in insulation resistance.