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INTERIM DEVELOPMENT REPORT
FOR
A POROUS SUBSTRATE RESISTOR AIMED AT MINIATURIZATION OF METAL-FILM RESISTORS

This report covers the period 1 January 1963 to 31 March 1963

Prepared by
Theodore Matley
Weston Instruments & Electronic Division
Daystrom Incorporated
Newark 14, N. J.

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Navy Department - Bureau of Ships - Electronics Division

Contract No. NObsr - 87542 Phase I
Project Serial No. SR-0080303, Sub-task 9636

WESTON INSTRUMENTS
Division of Daystrom, Incorporated, Newark 14, New Jersey

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TABLE OF CONTENT

Part I

Part II

Part III

ABSTRACT

Milestones △x

Porous ceramic substrate materials were evaluated in order to obtain a ceramic substrate material to replace the glass substrates. A ceramic substrate material was selected for use that is superior to any material that has been tried previously. △1a

Metallizing techniques have been further improved and a better understanding of the techniques has been obtained. △4

Physical parometers of the substrates were recorded and and attempt was made to correlate these parameters to resistance value. △2 △4

Terminals of various types were tested for use on the porous substrates. Some of the terminals were applied before metallizing and others were applied after metallizing. A method was incorporated for use in the project. △8

Methods of metallizing a substrate that has been cut from a porous disc are described and a metallizing procedure selected for use in the project.

Tests were conducted on heat treatment of the metallized resistors. Resistors were monitored to show changes in resistance value during the heat treating cycle. Effect on high resistance values were compared to the effect on the lower resistance values. △5

PART I

PURPOSE

The purpose of Phase 1 is to conduct a study of techniques and processes to determine the capability of Weston's proprietary chemical deposition process for producing metal film resistors on porous substrates. The study shall be directed toward the determination of the feasibility of using this process in developing the accurate, precision, metal film resistive element which can be adaptable to micro-miniature circuits. Such techniques as chemical deposition, etching, markings, etc. shall be investigated. Chemical concentrates, metallizing cycles, curing cycles, and other factors shall be determined. The effects of these factors on the resistivity per square, temperature coefficient and stability shall be determined.

Ten laboratory samples of various wattages (1/20w to 1/2w) in different configurations will be furnished. Electrical and environmental characteristics shall be determined on additional laboratory samples using Military Specification MIL-R-10509D as a guide. Specific wattage ratings, configurations and techniques shall be negotiated with the Bureau of Ships.

GENERAL FACTUAL DATA

Personnel: The names of the engineers and engineering aides together with the man hours spent on the project in this quarter are as follows:

Man-Hours

R. Drewes	34	E. Swain	75
T. Matley	503.5	T. Kropkowski	444
R. Smith	505	A. DeRogatis	4
R. Busch	394.5		

Patents: The inventions covered by any patent or patents issued as a result of U. S. patent application Serial #124716 filed 17 July 1961, for Electrical Components, and the patent application Serial #200, 461 filed 6 June 1962, for Electrical Resistors, are specifically excluded from the license grant of the Patent Rights Clause of this contract.

DETAILED FACTUAL DATA

Search for Ceramic Substrate Material △1a

During this period a new milestone was established. This milestone was designated △1a and was entitled, "Search for Ceramic Substrate Material." The reason for this action was the general agreement, by all concerned with the project, that the physical properties of glass are inferior to ceramic when used as a resistor substrate. Under this milestone a renewed effort will be made in an attempt to obtain a porous ceramic that could be used to replace the glass substrates. The glass substrates used until now were the only materials available in the porosities required by this project. The status on availability of porous ceramic substrates is detailed in Appendix A.

Two porous ceramic substrates of the necessary porosity can now be obtained. These are quartz and alumina. Physical properties (other than porosity) of the two (Appendix B.) show that they are equally desirable as a resistor substrate. Of the two ceramic materials, the quartz will be used because it has a greater uniformity of porosity. Theoretically, uniformity is achieved by sintering spherical ceramic particles of uniform diameter. Quartz particles can be obtained which are nearly spherical whereas the alumina particles have a shape that could be described as a hexagonal flake. Due to the shape uniformity the quartz particles can be more closely screened for size than alumina. This is an important parameter for producing a uniform substrate.

The size of the quartz particles largely determines the size of the pores after sintering whereas the alumina must be packed with a material which is removed during the sintering to help fix the porosity. It is evident that the quartz can be more readily controlled for pore size and uniformity than the alumina. Comparative uniformity of pore size can be shown by Chart A and Curve A on quartz and Chart B and Curve B on alumina. These charts and curves give a typical pore size distribution of the respective materials. From the charts, if a medium pore size of 65 micron is selected then the following data indicates the superiority of quartz over alumina in uniformity of pore size:

Material	Medium Pore	±5 of Medium	±15 of Medium
Quartz	65 micron	39% of pores	78% of pores
Alumina	65 micron	20 1/2% of pores	58 1/2% of pores

The quartz can be controlled even closer than is shown on the chart if needed. This would be done by closer control when screening. Differences in pore size from substrate to substrate usually occur but the material that will be purchased for this project will be individually selected. A parameter that may prove useful for selection is an air flow test which is conducted by the vendor. A sample report on twelve 2 1/2 inch discs is shown in Appendix C.

It can also be noted that the use of quartz, which has been available only during the last few weeks of this period, shows a much better uniformity of pore size than the glass used previously. This observation is based on visual inspection and on comparison of uniformity of resistance readings (Appendix K) which is reported in a subsequent section.

Method of Solution Application - Substrate Discs 4

During the period covered by the second report, a number of metallizing methods were evaluated. The best method devised at that time was as follows:

1. Sensitized by gasketing the porous disc within an acrylic chamber and passing sensitizing solution slowly thru the substrate disc by raising and lowering funnels to control the speed and direction of flow of the sensitizing solution.
2. Metallize with a slow solution flow using a similiarly gasketed acrylic housing as used in sensitizing. The speed of solution flow is controlled by raising or lowering a reservoir (a separatory funnel) thereby varying the head of the solution.

Use of this method gave marked improvement in the percentage of substrates that were metallized successfully as determined by visual inspection. The move to the new metallizing technique desscribed above was made simultaneously with the new sensitizing technique. In order to ascertain whether the improvement in metallizing was due to the combination of changes or due to one, a number of substrates were metallized retaining the sensitizing technique. The substrates were metallized in a Buchner funnel. The quality of the metallized substrates appeared to be as good as when the substrates were metallized in the acrylic housing. Good metallizing is dependent on good sensitizing. No method of metallizing will give good results if the substrate has not been properly sensitized. Proper sensitizing of the substrate requires a slow flow of sensitizing solution with care exercised to ensure total coverage or saturation of the substrate. The sensitizing is followed by a vigorous rinse to ensure removal of excess sensitizing solution. The substrate is then metallized but temperature control of the metallizing solution is important in order to control the resistance value.

Effect of Substrate Parameters on Resistance △2 △4

Resistors that were made during the first two periods of the project show no evidence of correlation between resistance value and the porosity of the glass substrates as rated by the vendors. Visual inspection of hundreds of glass substrates of various porosities indicated that the vendors' ratings of porosity does not give a proper indication of the internal surface area. The internal surface area is the parameter that would exercise the greatest effect on the final resistance value of a substrate in a controlled metallizing process. The reason that the glass substrates vary so greatly in uniformity of internal surface area is that the vendors' prime interest in manufacturing the disc is for its use as a filter rather than for the internal surface area. As a filter, its purpose is to retain particles of a minimum size with no effort exercised to reproduce the number or density of pores. Many of the glass substrates appear to be over fired with subsequent settling of particles and closing of pores.

During the third period, an attempt was made to classify the glass substrate discs and to preselect the substrates for uniformity of parameters which would allow resistance values of the metallized substrates to be duplicated.

The following parameters were to be controlled or tested in order to ascertain which ones can be related to the resistance of the substrate:

1. Vendor designation of pore size
2. Grind substrates to uniform thickness
3. Weigh the discs after grinding. This weight would be directly proportional to the density as the sizes of the substrate will be identical at this point. This would give a function of voids to solid ratio of the substrate.
4. Check solution flow. This is done by checking rate of water flow through the substrate or measuring the amount of metallizing solution that has flowed through the substrate at a fixed time. (If two parts show the same density but different flow rates, it would be indicative of closed pores in the slower flowing disc if the pores are of the same size.)
5. After the parts are metallized, resistance measurements will be taken on the disc using a device to read through the disc and another that would take surface readings.

Four Corning coarse glass discs were ground down to uniform thickness and weighed. The weights were recorded and a large separatory funnel of water (2432 cc.) was connected by tubing to a housing within which the substrate was gasketed. At a fixed head and at a fixed vacuum the water was allowed to pass through the substrates and the duration of the flow checked (2 times) with a stop watch. The results of this test are recorded in Appendix D. The results of the test show that the flow rate could not be predicted by the weight or density of the substrate. The lightest density did not have the fastest flow nor did the highest density have the slowest flow rate. The two intermediate weight parts that were nearly identical in weight gave both the fastest and slowest flow rates.

The above tests were repeated using Corning extra coarse substrates. The results of this test are tabulated in Appendix E. This test involved glass substrate with a wide range of weights or densities. The lightest substrate had an extremely low density when compared to the others and did exhibit the fastest flow rate but not to the degree that its density would indicate. The other substrates did not exhibit any better correlation than the previous test.

Some additional substrates were ground, weighed and then metallized. The flow of the metallizing solution was not accelerated by the use of vacuum. Metallizing was done by using the separatory funnel and acrylic housing. Flow rate was determined by recording the amount of metallizing solution that flowed through the substrate in a fixed period of time. (In this test 100 seconds). The results are tabulated in Appendix F. After heat treating the parts were measured for resistance value as shown. The reading through the substrate is a better reading to compare for internal resistance as the other readings are surface readings. This test, as in the previous test, shows no correlation between density and solution flow. There also appears to be no correlation between density and resistance nor is there any correlation between solution flow and resistance. Lack of correlation between density and resistance is indicative of non-uniformity due to closed pores or voids. Lack of correlation between solution flow and resistance value indicates irregular porosity and non-uniformity of pores. The validity of the above test is dependant upon accurate resistance measurements.

The resistance measurements were questionable, therefore the resistance measuring devices were subjected to a series of tests. Using a number of metallized substrates the devices were checked for reproducibility of readings by rotating the substrate within the device taking a resistance measurement after each move of the substrate until a series of ten readings were taken on the substrates. The extremes of the readings are listed in Appendix G. No particular mean resistance could be established. Another test was performed whereby some metallized discs were cut into sizes that measured .16" x .16" x .70". The individual resistors were measured for resistance value from one surface to the other (a through reading thickness) and the value recorded. Terminals of silver paint were then applied on the ends and a resistance measurement taken. The readings taken with terminals, represent values of finished resistors. The readings were compared to the "through" readings (Appendix H). No apparent correlation can be established between resistance readings taken on the substrate film and resistance readings taken on the resistor when a terminal is in place. Evidently any touch or probe contact with the film itself give a very erratic reading due to high resistance contacts.

In order to be able to ascertain success of metal deposition and evaluate substrate material and uniformity by resistance measurements rather than visual inspection, the terminal must be affixed to the substrate before metallizing. This requires the resistor to be cut to the ultimate size before metallizing so that the terminals can be fixed in place. This report subsequently will show how this was done, thereby eliminating the need for the disc reading devices.

Terminals 8

Terminals of conductive epoxy silver had been used in the first two periods of this project. This terminal was used on a temporary basis to enable resistance measurements to be made and it was understood that development work on terminals would be undertaken. The shortcomings of the epoxy silver terminals were enumerated in the report of the second period or quarter.

The following terminals were listed for evaluation during this period for use on the porous substrate resistor:

1. vapor deposited metal
2. sputtered metal
3. sprayed metal
4. dental amalgams
5. fired metal frit

Glass substrates were cut to .16" x .16" x .70" and terminals were applied at the ends. Ultrasonic cavitation in water was used to ascertain the degree of adhesion of the terminal to the substrate. Gold was vapor deposited[1] on a substrate and tested for adhesion by ultrasonic cavitation in water. The gold flaked off readily. Because

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[1] Glass substrate was masked and vapor deposited using an R. C. A. EMV-1 Evaporator Unit. Gold wire was evaporated for 75 seconds at a current of 24 amps on each end.

sputtering was expected to give the same poor results, no effort was expended to prepare masks and a sputtering chamber. Additional substrates were sprayed with copper and tested.[1] The adhesion of the copper to the substrate was also very poor. The company that sprayed the copper indicated that they could probably improve the copper adhesion if the substrate had been sprayed while hot. Sprayed copper terminals will be tabled for the present, but can be reopened if other terminal methods do not prove to be successful.

Dental amalgams were considered for a number of reasons. They can be applied to the substrate without the need of heat and therefore, can be applied after the film is in place if this technique is required. Special formulations can expand and others contract while hardening. This feature could be of advantage in obtaining adhesion within the pores thereby resulting in a strong terminal.

The first dental amalgam that was tried had the following composition:

Silver	34.9 gms	69.7%
Tin	13.0 gms	26.1%
Copper	2.1 gms	4.2%
	50.0 gms	100.0%

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[1] Copper spraying was done by Metco Company of Westbury, Long Island, New York.

The metal powders listed were mixed and amalgamated with mercury. The copper did not appear to amalgamate properly. Mercury is added to the powder in a ratio of 5 parts powder to 6-8 parts mercury. The excess mercury, if any, can be drained off. The above mix was tried four different ways:

1. Applied with spatula to ends of substrate as mixed.
2. Excess mercury drained off and the remainder applied to substrate.
3. A small amount of zinc was added as a scavanger. The amount is so small that it does not noticeably amalgamate any excess mercury but acts as a reducing agent so that the other metals are not oxidized.
4. Same as (1) except the amalgam was allowed to set in the mortar for one hour before it was applied to the substrate. The methods tried took an unusually long time to harden. After becoming hard (24 hours later), a test for adhesion gave very poor results.

Additional copper was added to the amalgam. Copper should increase strength and shorten the hardening time. The results were still poor because the copper did not amalgamate properly. In order to improve the above listed alloy, the metal powders could be melted into an ingot and the ingot then powdered before the addition of mercury. The facilities for preparing and powdering an alloy are not available so a powdered alloy has been ordered from L. D. Caulk Company, Newark, N. J., but has not as yet been received. An alloy of 1 part zinc to 2.85 parts silver, was prepared and amalgamated. This terminal gave good strength. A pull test of 7.5 lbs (Tensile) was required to break the bond. This amalgam was rejected because of its poor temperature limitation of 260° F. Another amalgam of tin-mercury was tried but the results were very poor. Additional work may be attempted with amalgams when the alloy powder is received if a better terminal cannot be obtained using another method.

The metal and glass frit terminal received more effort than the other methods of termination that have been described. This method of termination is universally accepted in the electronic industry and the engineers at Weston have had some experience with its use and have found it acceptable in this type of application.

The metal frit terminals require that the terminals be affixed to the substrate prior to metallizing. The high temperatures of 1000°F and higher that are required to cure or fire the metal frit onto the substrate would destroy the metal resistive film if the terminals were fired after metal deposition. In order to place the terminals on before metallizing the substrate will have to be cut to size before terminating and therefore, before metallizing.

A platinum-gold paste was the first of the metal-frit materials that was tried. Dupont #7553[1] was applied to the ends of the substrate and fired at 1100°F for one hour. The terminals looked good but ultra-

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[1] E. I. DuPont de Nemours & Company, Inc., Electrochemicals Dept. Wilmington, Delaware

sonic cavitation in water caused some particles to flake off. A liquid gold from Engelhard Industries Inc., was tried but no better results were obtained. These materials may have been shelf aged or contaminated so a new material was obtained.

Ceronics Inc., of Matawan, indicated a willingness to work with us to obtain the proper metal frit to solve the terminal problem. They supplied a palladium-gold paste (#1212) and a number of glass substrates were terminated and fired at 1050°F for 15 minutes with this material. Some of the terminals gave good results when tested for adhesion and others did not. An experiment was run to determine which section of the furnace gave the best firing results. Substrates fired in this zone subsequently gave good results when tested for adhesion. The glass substrates were metallized. A high resistance area (poor metal film deposition) occurred next to the terminals on some glass substrates. This may be due to incomplete firing of the terminal material which may contaminate the substrate next to the terminal thereby inhibiting the metal deposition or the terminal may have masked off or retarded the solution flow thereby causing lean deposition of metal film near the terminals.

In order to check any effect the terminal may have on the resistance flow near the terminal, an experiment was run using glass slides that measured 1" wide by 2 1/4" long. A terminal was fired on each end to a depth of approximately 1/4". Metal resistance readings taken with probe and ohmmeter to check for uniform resistance and for any high resistance area adjacent to the terminals. A sketch and resistance readings of three glass slides are shown in Appendix I. No apparent high resistance band exists in the film next to the terminals based on visual inspection and resistance measurements taken on the glass slides. Although the high resistance band of metal film is not apparent on the glass slides, the high resistance bands were still noticeable on the glass substrate parts both visually and by resistance measurement. Appendix J. shows measurements taken at the terminals and other measurements taken just within the terminals. A significant drop in resistance value on readings taken just within the terminals indicates a high resistance band or area. Resistance measurements taken just inside the terminals normally read higher because of the poor high resistance contact made with the probes and this normally offsets the small drop in resistance expected because the probes are closer together than the terminals.

At this time a porous ceramic (Filtros Quartz) was obtained and some substrates were cut from the material. The sizes cut were . 16" x . 16" x . 70" which is the same as the glass substrates that were used in the preceding tests. The terminals (Ceronics #1212 - the palladium gold frit) were fired onto the ceramic substrate in the same way and using the same firing cycle as on the glass substrates. These parts were then metallized and checked for a high resistance band next to the terminal. The high resistance band was not visible and the improved appearance of the metallizing on the ceramic over the glass substrate was very striking. Resistance measurements through the terminals and just within the terminals were made on these parts as on those in Appendix J. The readings of the ceramic resistors are shown in Appendix K. Appendix K indicates that, although there are exceptions the terminals used with ceramic substrate showed a marked improvement over the glass substrate in regard to a high resistance band. With the ceramic substrate the great drop in resistance values just within the terminals does not exist to any degree when compared to the glass substrates.

An attempt was made to fire a lead wire directly onto the ceramic substrate with the use of the palladium gold frit but this attempt was unsuccessful. Additional resistors will be made using the metal frit terminals to further evaluate this method of termination.

Metallizing of Cut Substrates

The need for placing a terminal onto the substrate prior to metallizing was explained in the previous section. Because of this change the substrates had to be cut to resistor size prior to terminating and therfore, prior to metallizing. A nominal size of resistor, therefore, had to be selected. The size of the cut substrate that was selected was .16" x .16" in cross section by .70" in length. This size was selected because it could fit into a transfer mold that molds the epoxy coating over the conventional 1/2 watt resistor. Should the porous resistor lend itself to this type of encapsulation, a transfer mold will be readily available.

Rubber gaskets were made that could hold three of the cut substrates simultaneously within the sensitizing fixture (2 funnel) or the Buchner funnel. A number of resistors were produced using this method. The first substrates were of glass and the ones done later were of ceramic. The cut glass substrates gave the same poor results that had been obtained earlier with the discs. This is undoubtedly due to the inherent poor qualities of the glass as described earlier. Column 1 in Appendix L, shows the resistance values of glass resistors. The resistance value is given in groups of three. The three resistors were sensitized and metallized simultaneously and each group of three is from the same disc. The only variable possible other than the non-uniformity of the substrate is the terminal. (This also is true of Column 2 yet it shows an improvement and Column 3 which appears to be the best.)

The resistance value from group to group is not significant. Comparisons between Column 1 and Column 2 indicates that the ceramic tends to have a more uniform composition.

Because there was some evidence that the cut substrates when gasketed would not allow metallizing to proceed on the sides that contact the gasket, immersion metallizing was again attempted. This had been tried earlier but it had failed. The earlier parts were of cut glass and therefore, not uniform which may not have allowed the solution to penetrate. The sensitizing technique had also been improved. The cut ceramic substrates were sensitized by gasketing using the funnel technique then transferred to a beaker of metallizing solution under a bell jar so that vacuum could be used. This was done in groups of three. The results are shown in Appendix L, Column 3. The groups of three tend to group somewhat better than the previous columns. Additional data will be compiled on this technique because it has the advantage of allowing many resistors to be run simultaneously. Resistors broken in half and observed under a microscope show metal deposition throughout.

Heat Treatment △5

After the metal film deposition process the metal resistive film must undergo heat treatment in order to stabilize the resistor. Three important changes occur during the heat treatment. These are as follows:

1. The resistance of the resistor changes. This occurs in two stages. In the first stage there is a drop in resistance. This generally occurs in the first 15 minutes of heat treatment. The percentage drop appears to be greater in the lower resistance value than those of high resistance values. During the second stage the resistance value of the resistor increases. In the higher value resistors this increase is greater than those of low resistance value. This increase in resistance or second stage generally occurs over a period of hours rather than minutes as in the first stage. The temperature of the heat treatment will have an effect on especially the second stage.

2. The second effect of the heat treatment is on temperature coefficient of resistance. This is closely related to the temperature that is selected.

3. The third effect of the heat treatment is stability. This is a function of temperature and time. Length of time of heat treatment appears to be quite important. A temperature selected for temperature coefficient properties yet too low

for good stability may still be used to obtain good stability by extending the period of time of the heat treatment.

Based on previous experience, a temperature of 500°F for 15 hours was selected for the initial heat treating experiments. A number of resistors were monitored for resistance value during heat treatment by the use of a recorder. The resistors that were monitored range in resistance value from 75K to 3.95K immediately after metallizing. Graph A shows the change that occurs in resistance value of the resistors in the first few minutes. The 74K resistor only dropped 27% in resistance value before it started to increase in resistance value. At 10 minutes the resistor again read 74K and at 15 minutes was 50% higher in resistance value than when it had started. The 74K resistor eventually reached approximately 2 megohms in value at the end of 15 hours.

The low resistor that measured 3.95K after metallizing lost 86% of its resistance value in the first 10 minutes. Graph B shows that this low resistor increased very slowly in resistance value after the initial drop. After 13 hours this resistor still retained a drop of 77% in resistance value. The sudden increase in resistance value at the end of the curve is due to a temperature coefficient effect. The resistors were returned to room temperature from 500°F. The curves indicate that all of the resistors under test exhibited a negative temperature coefficient and that the lower value resistor had a smaller temperature coefficient than the higher value resistors. A number of other resistors were heat treated at 500°F for 15 hours. These resistors were not monitored during heat treatment. An R_1 (reading after metallizing) was taken and an R_2 (resistance value reading after heat treatment). The resistance readings are shown on Graph C.

Analysis of the graphs leads to the following possibilities:

1. The initial drop in resistance is due to the annealing or stress relief of the metal film. If this is true, then the thicker films (lower value resistor) would tend to be more stressed than the thinner films (high value resistors) and therefore, show a greater resistance drop in the initial stages.
2. The second stage of heat treatment is due to oxidation of the film surface and causes an increase in resistance. This effect would be more noticeable in the thinner films (higher value resistor) for proportionately more of the thickness of the metal film would be affected than in the heavier metal film (low value resistor.)

Past experience with this metal film indicates that one heat treating cycle is not sufficient for a range of resistance values. The resistors must be segregated after metallizing and placed in smaller groups. Each small group must then be heat treated with a cycle that brings out the optimum of stability and temperature coefficient. Graph B indicates that the heat treatment gave the 3.95K (initial resistance value approximately 900Ω final value) the best temperature coefficient of the resistors tested. The graph indicates that the heat treating cycle

would have imparted an even better temperature coefficient to a lower value resistor. Tests of this type must be conducted in order to obtain the best temperature coefficient and stability for various resistance ranges.

APPENDIX A

	Vendor Contacted	Comment
	American Lava Corporation Chattanooga, Tennesee	Currently setting up project in Lab. Feel that they can supply samples of new body blended especially for this project. Samples in 8 - 9 weeks.
	Aluminum Company of America Pittsburgh, Pennsylvania	Not available
	Centralab Electronics Division Globe Union Inc. Milwaukee, Wisconsin	Manager of Research and Development was in and since has supplied samples of Alumina in two porosities.
	United States Stoneware Co. Akron, Ohio	Presently do not manufacture ceramic in the porosities required.
	Coors Porcelain Company Golden, Colorado	Presently do not manufacture ceramics over 15 micron pore size.
	Thor Ceramic Bloomfield, New Jersey	Not available
	Saxonburg Ceramics Inc. Saxonburg, Pennsylvania	Not available
	National Ceramic Trenton, New Jersey	Not available
	Lab Equipment Company St. Josephs, Michigan	Not available
	Selas Flotronics Spring House, Pennsylvania	One porous alumina available within the range required by this project.
*	Norton Company Worcester, Massachusetts	A range of pore sizes are available but only in Alumina
*	Filtros Incorporated East Rochester, New York	A range of pore sizes are available in Quartz and Alumina

* A trip was made to the above companies to discuss our requirements.

APPENDIX B

	Fused Alumina	Boro Silicate Glass	Silica (Quartz)
Fusion Point °F	3500 *		3050
Load R., Temp °F for 10% shrink at 25 lbs	2800 *		3000 *
Thermal Expansion °C	7.0×10^{-6} **	3.3×10^{-6} *	5.9×10^{-7} *
Thermal Conductivity cal/sec. cm. °C.	400×10^{-4} **	$27. \times 10^{-4}$ *	$19 - 20 \times 10^{-4}$ *
Softening Point	2624°F **	1290 *	2,650°F *

* Langes - Handbook of Chemistry

** American Lava Corporation - AIS. Mag Ceramic Chart #611

APPENDIX C

			AIR FLOW at 2"H_2O	
			CFM	
QF-SF-1	WGT. (g.)	THICKNESS (in.)	Per Disc	Per ft.^{2}
A	75.1	0.51	0.84	24.7
B	74.2	0.51	0.89	26.2
C	71.6	0.50	0.95	27.9
D	72.3	0.50	0.87	25.6
E	73.2	0.51	1.01	29.7
F	71.9	0.50	0.96	28.2

			AIR FLOW AT 10"H_2O	
			CFM	
QF-SF-2			Per Disc	Per ft.^{2}
G	58.6	0.49	0.87	25.6
H	60.4	0.49	0.79	23.2
I	62.1	0.50	0.77	22.6
J	60.3	0.49	0.88	25.9
K	62.0	0.50	0.75	22.0
L	63.4	0.51	0.80	23.5

Filtros, Inc.,
Ref: 20-35, 36

APPENDIX D

Substrate #	Material	Thickness	Weight	Flow Rate (2432 cc H_2O at 100 mm Hg)
2	Corning Coarse 40-60 micron pore	115"	3.4110 gms.	73.5 73.7 sec.
3	"	115"	3.3206 gms	74.5 75.6 sec.
6	"	.115"	3.4140 gms	78.0 80.3 sec.
7	"	.115"	3.5008 gms	76.6 77.0 sec.

APPENDIX E

Substrate #	Material	Thickness	Weight	Flow Rate (Aug.) (2432 cc. H_2O at 100 mm Hg)
1	Corning Extra Coarse 175-220 micron pore	.114"	3.0988 gm	69.6 sec.
2	"	.115"	2.5232 gm	65.7 sec.
3	"	.114"	3.3720 gm	71.2 sec.
4	"	.114"	3.4804 gm	69.7 sec.

APPENDIX F

Substrate #	Material	Thickness	Weight	Solution Flow	Resistance		
				(100 sec.)	T*	B**	C**
1	Corning Coarse 40-60 micron pore	.115"	3.1460 gms	525 cc.	12KΩ	50Ω	1 mg.
5	"	.115"	3.7400 gms	475 cc.	340Ω	200Ω	300Ω
8	"	.114"	3.1950 gms	455 cc.	4KΩ	340Ω	70KΩ
9	"	.114"	3.1000 gms	430 cc.	70Ω	30Ω	60KΩ
11	"	.114"	3.4720 gms	370 cc.	500Ω	900Ω	2K
12	"	.112"	3.0006 gms	590 cc.	111Ω	50K	3.8K
13	"	.113"	3.3838 gms	380 cc.	220Ω	650Ω	12K

* Reading through thickness of substrate

** Surface reading - one reading for each surface

APPENDIX G

Range of Resistance Readings on Metallized Substrates using Devices

Resistance range - in 10 readings

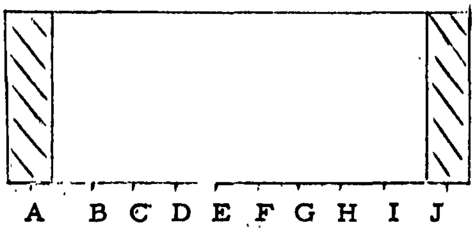
Substrate	Through-A side-up	Through-B side-up	Surface A side	Surface B side
11	43-80 Ω	24-46 Ω	.37-1.2 KΩ	.3-10 KΩ
13	100-300 Ω	110-250 Ω	1.1-100 KΩ	.58-5 KΩ
14	160-220 Ω	100-225 Ω	1.5-10 KΩ	135-4,000 KΩ
15	8.2-540KΩ	10.2-30 KΩ	1.2-250KΩ	∝
16	80-500KΩ	1.7-500KΩ	1-22 KΩ	1.3 Meg.
17	500Ω-4.2KΩ	.3-2.8KΩ	35-100 Ω	40 KΩ ∝
18	3KΩ-1.1 MegΩ	.56-35 KΩ	1.5-5.4 KΩ	50 Meg. ∝

APPENDIX H

Resistor #	Resistance (Fixture Through)	Resistance Terminal Ag. Paint	Material
28a	3.8-6.5KΩ	80K	Corning Coarse 40-60μ pore
b	85-500KΩ	100K	"
c	44-70 KΩ	17K	"
d	38-40 Ω	380Ω	"
e	85-95 Ω	230Ω	"
f	1KΩ	1.2K	"
29g	32Ω	280Ω	Ace B - 70-100 μ pore
h	27-31Ω	430Ω	"
i	1-1.2KΩ	600Ω	"
j	135-148Ω	1.3KΩ	"
k	2.3-2.7KΩ	1.25KΩ	"
l	21-35 KΩ	750Ω	"
m	10-32 KΩ	1.9KΩ	"
n	2.4-2.6KΩ	2.1KΩ	"
o	180-200KΩ	16KΩ	"
p	110-220KΩ	400KΩ	"
q	72-82KΩ	300KΩ	"
r	100-108KΩ	170KΩ	"
s	54-58 KΩ	500KΩ	"
t	21-24 KΩ	96KΩ	"
u	70-100KΩ	120KΩ	"
30a	950-1200Ω	6.5KΩ	Corning Coarse 40-60μ pore
b	1.5-1.7KΩ	5.5KΩ	"
c	85-92 Ω	1.6KΩ	"

APPENDIX I

Terminals

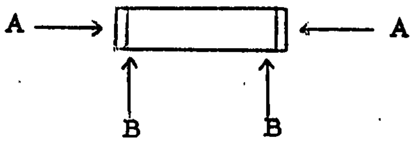


Points*	Slide #1	Slide #2	Slide #3
A-B	55Ω	105Ω	90Ω
A-C	58	107	90
A-D	61	115	92
A-E	69	127	100
A-F	74	145	105
A-G	81	150	112
A-H	84	150	120
A-I	90	160	125
A-J	63	145	129
B-I	85	100	110
J-B	75	115	115

No apparent high resistance band of metal film exists next to the terminals otherwise the A to J reading would increase remarkably over the A to I reading.

* Taken with approximately 1/8" rod laid across width at point indicated.

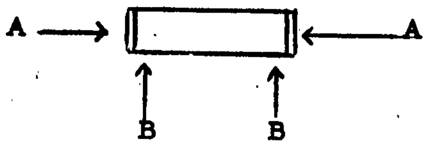
APPENDIX J



Glass Substrates

	Resistance	Reading
Part #	AA	BB
N	17K	2.2K
O	72K	900Ω
P	30K	3.6K
R	49K	1.8K
S	18.5K	1.5K
48 A1	92K	700Ω
2	16K	1.3K
3	45K	2.0K
48 D1	80K	1.5K
2	5K	3.4K
3	22K	9.3K
47 A1	13K	840Ω
2	83K	1020Ω
3	18K	1.5K

APPENDIX K



Ceramic Substrates

Part #		Resistance AA	Readings BB
48A	1	9K	1.5K
	2	4.8K	5.8K
	3	3.2K	4.2K
49B	1	1 Meg.	1.3 Meg.
	2	290K	300K
	3	300K	500K
	4	17K	21K
	5	10K	12.5K
	6	16.5K	19K
49C	10	4.4K	4.2K
	11	2.9K	15K
49D	2	5.2K	3.2K
	3	3.6K	2.2K
49A	5	67K	75K
	6	105K	65K

APPENDIX L

Resistance Readings

	1.	2.	3.
Metallize:	Buchner	Buchner	Immersion
Material:	Glass	Ceramic (Med)	Ceramic (Med)
	420K	9K	1.6K
	580K	4.8K	1.7K
	330K	3.2K	1.3K
	1.22K	17K	3.6K
	6K	10K	4K
	16K	16.5K	2.9K
	4.6K	820Ω	2.3K
	7K	820Ω	3.6K
	11K	1220Ω	13.5K
	41K	740Ω	90Ω
	22K	600Ω	85Ω
	500K	1100Ω	78Ω
	580K	600Ω	2.2K
	24K	830Ω	1.7K
	300K	1000Ω	2.2K
	120K		600Ω
	80K		500Ω
	270K		500Ω
	20K		1.7K
	54K		1.0K
	170K		1.2K

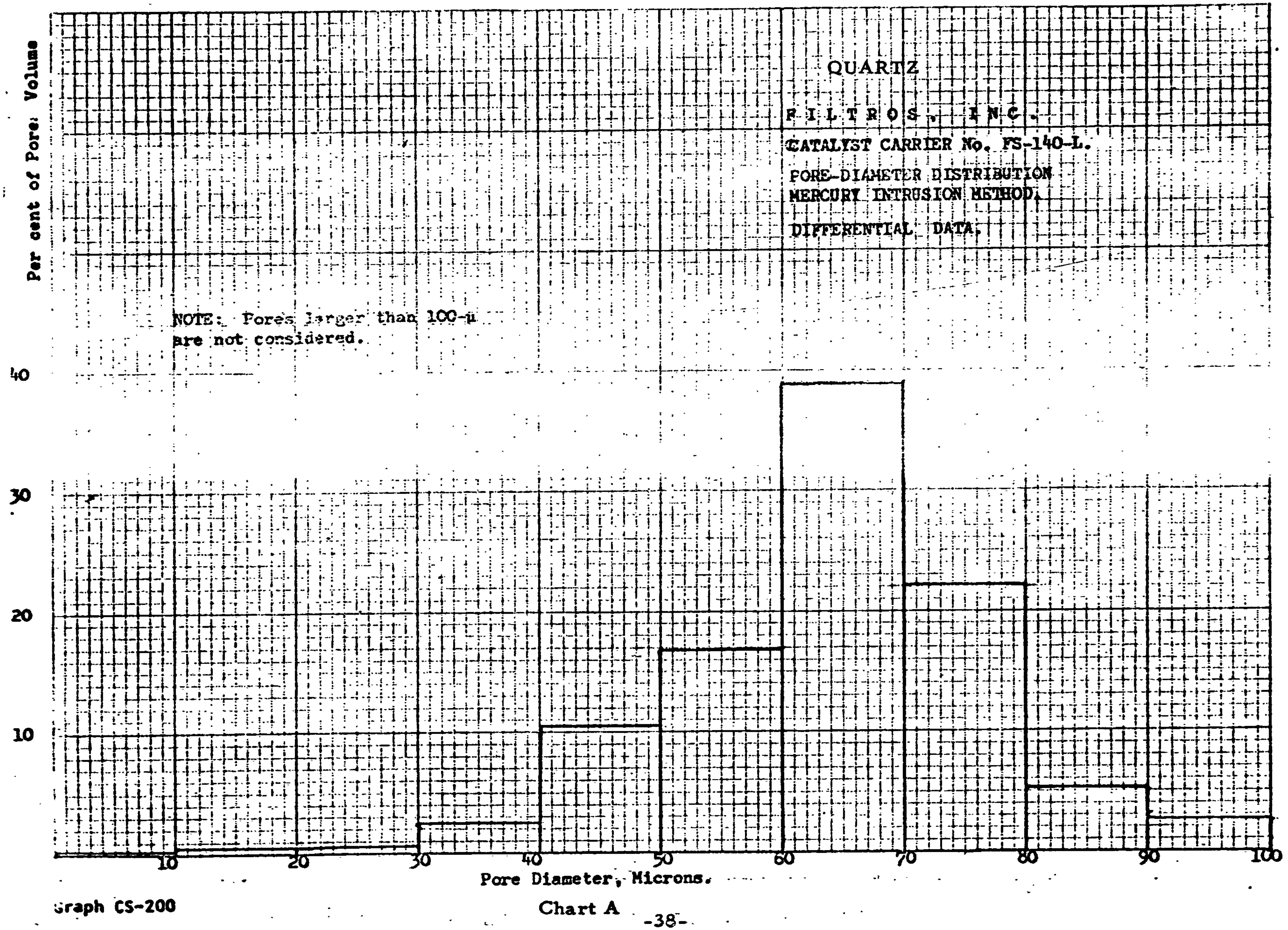
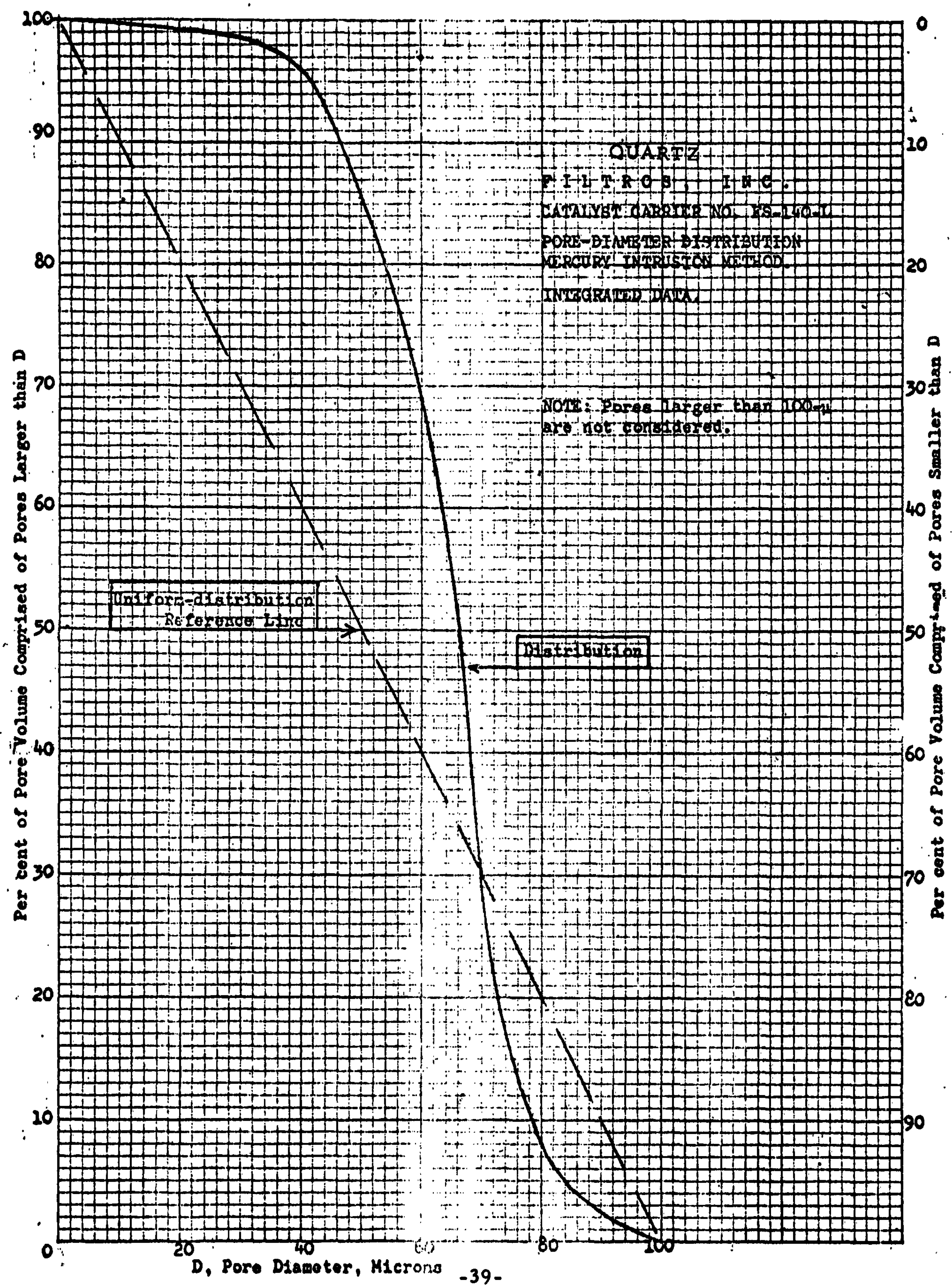
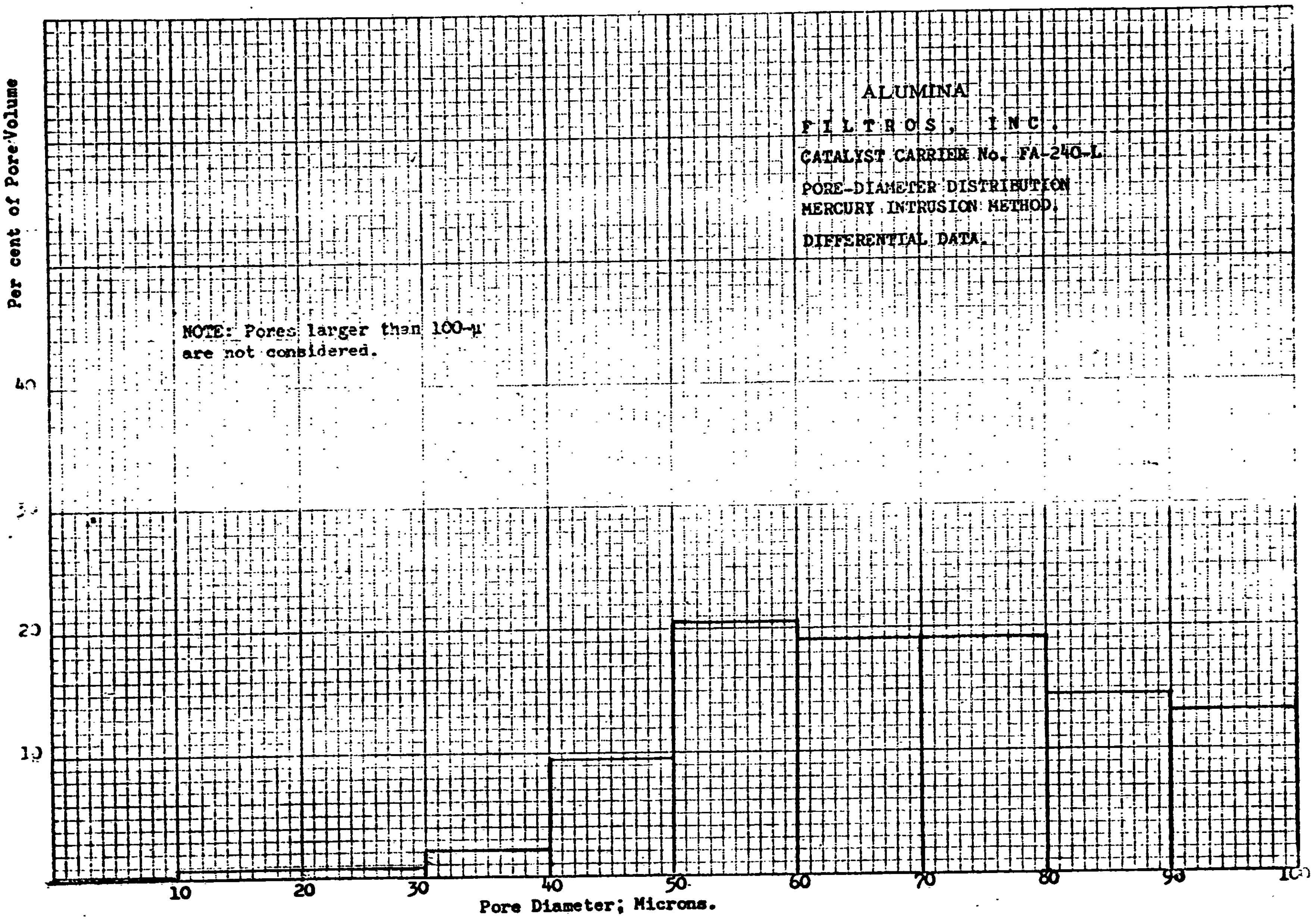

QUARTZ
FILTROS, INC.
CATALYST CARRIER No. FS-140-L.
PORE-DIAMETER DISTRIBUTION
MERCURY INTRUSION METHOD.
DIFFERENTIAL DATA.
NOTE: Pores larger than 100-µ
are not considered.
Per cent of Pore: Volume
40
30
20
10
10
20
30
40
50
60
70
80
90
100
Pore Diameter, Microns.
Graph CS-200

Chart A

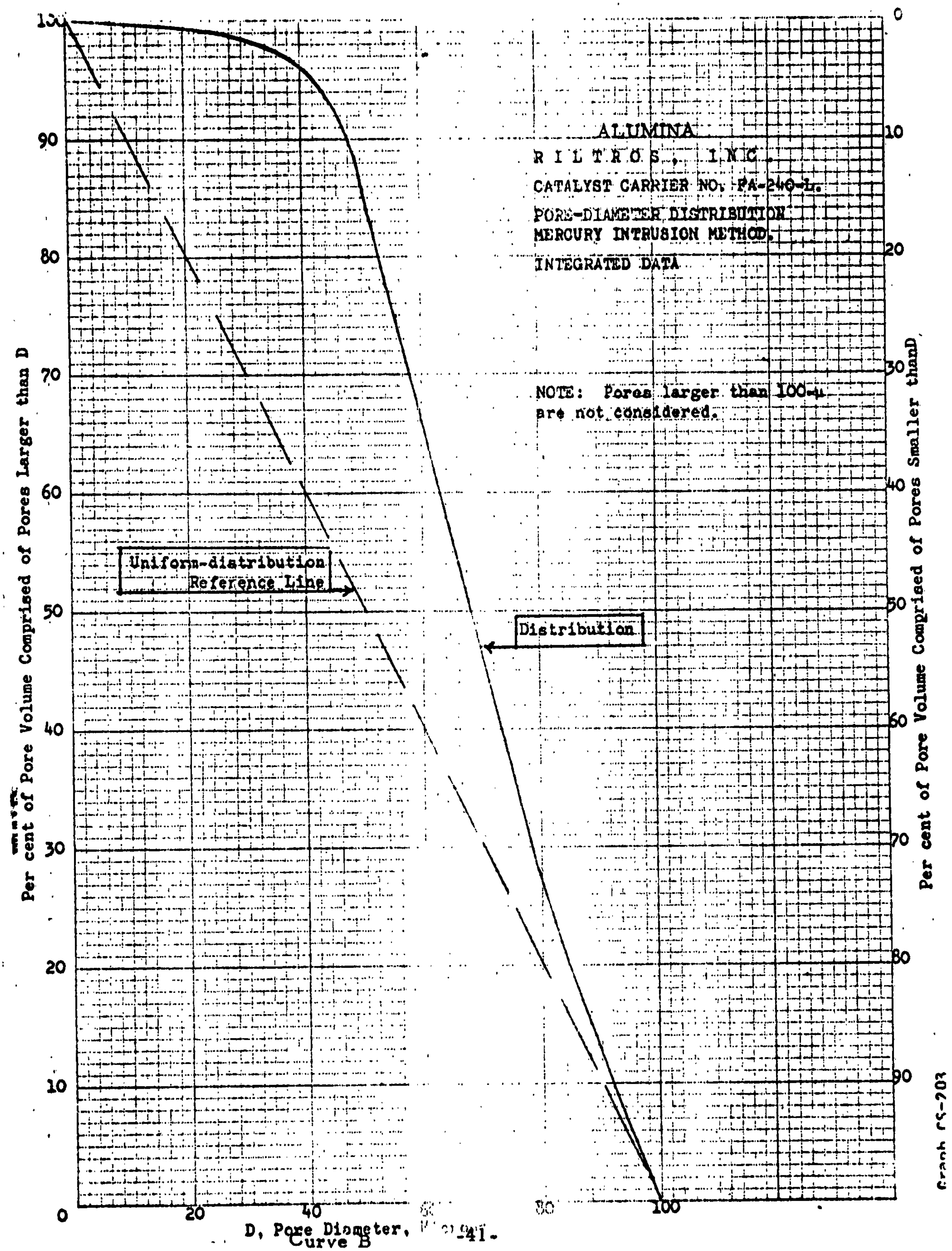

QUARTZ
FILTROS, INC.
CATALYST CARRIER NO. FS-140-L
PORE-DIAMETER DISTRIBUTION
MERCURY INTRUSION METHOD.
INTEGRATED DATA.
NOTE: Pores larger than 100-μ are not considered.
Uniform-distribution Reference Line
Distribution
Per cent of Pore Volume Comprised of Pores Larger than D
Per cent of Pore Volume Comprised of Pores Smaller than D
D, Pore Diameter, Microns
100
90
80
70
60
50
40
30
20
10
0
0
10
20
30
40
50
60
70
80
90
0
20
40
60
80
100

Curve A

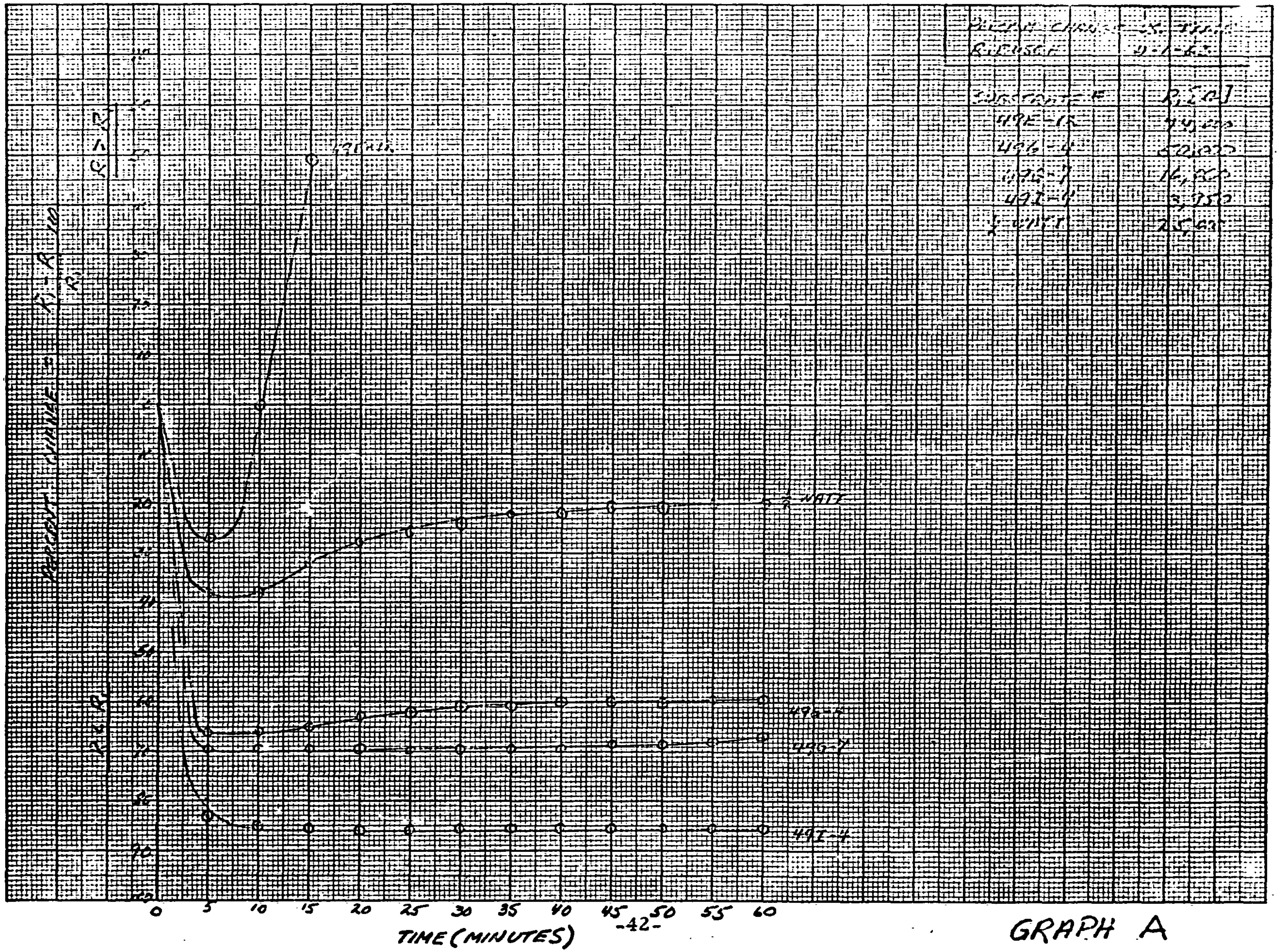


Graph CS-202

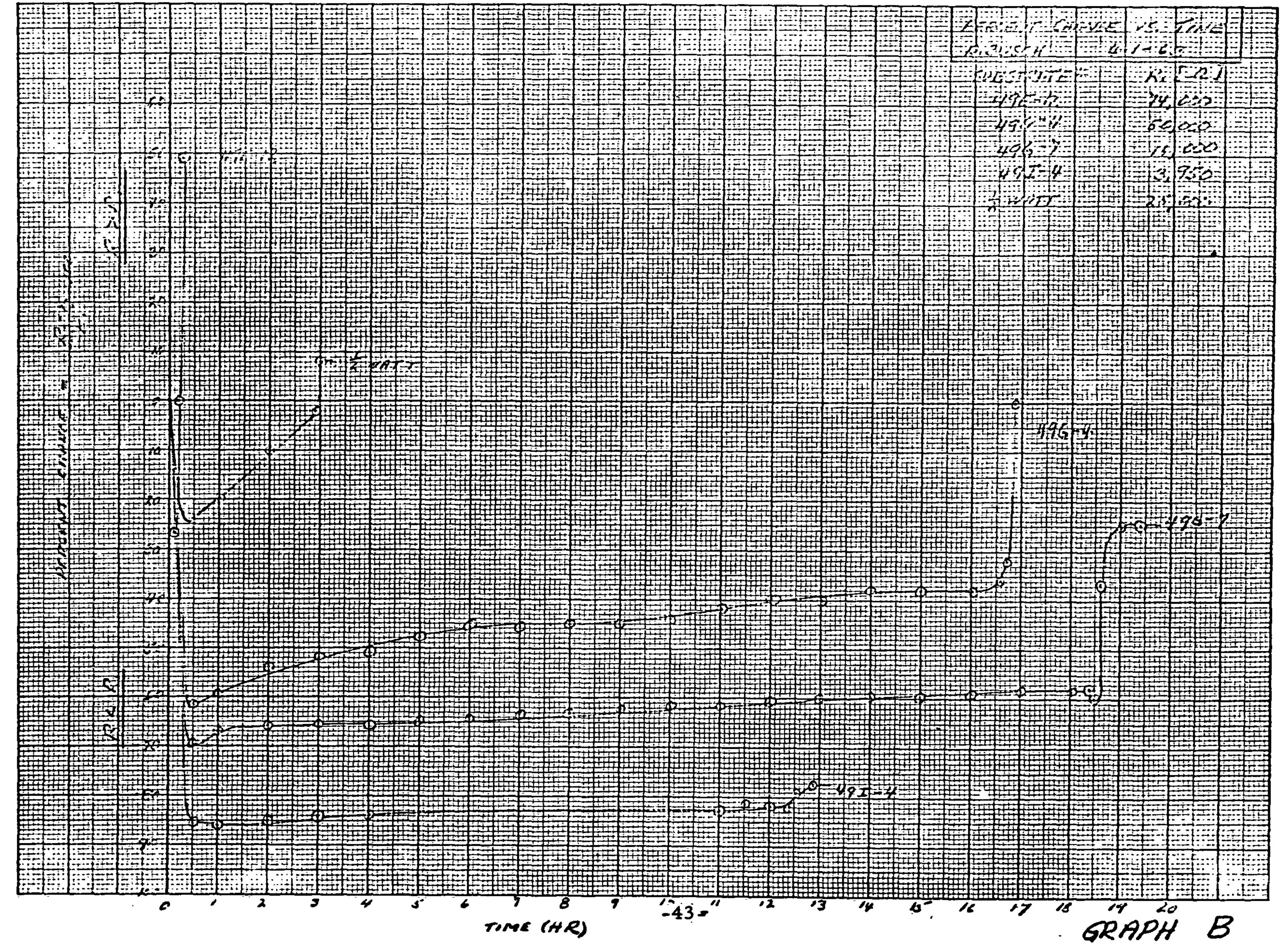
Chart B


ALUMINA
RILTROS, INC.
CATALYST CARRIER NO. FA-240-L.
PORE-DIAMETER DISTRIBUTION
MERCURY INTRUSION METHOD.
INTEGRATED DATA
NOTE: Pores larger than 100-μ are not considered.
Uniform-distribution Reference Line
Distribution
Per cent of Pore Volume Comprised of Pores Larger than D
Per cent of Pore Volume Comprised of Pores Smaller than D
D, Pore Diameter,
100
90
80
70
60
50
40
30
20
10
0
20
40
60
80
100
Graph CS-203

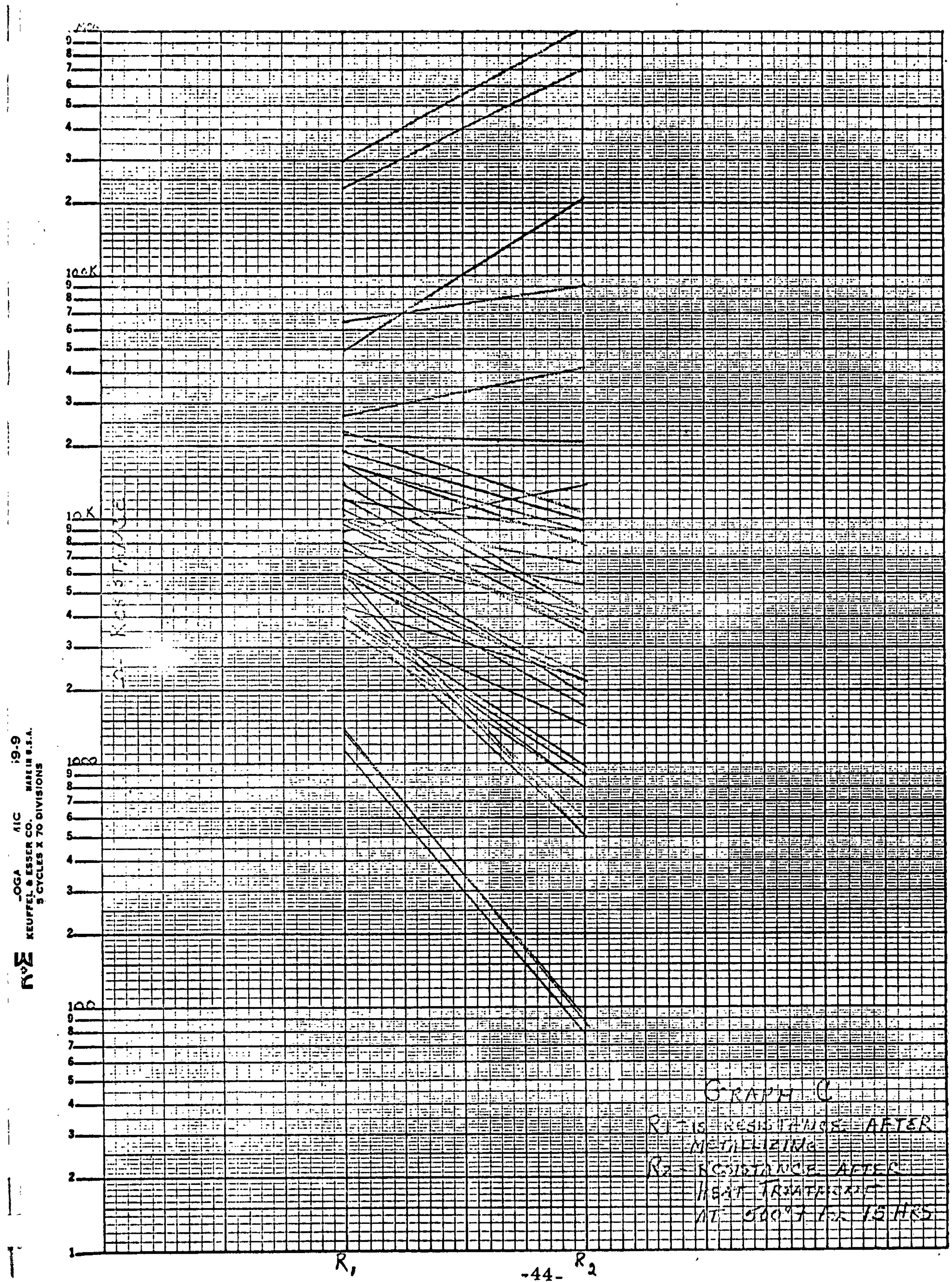
Curve B


PERCENT CHANGE vs. TIME
9-1-63
SUBSTRATE #
492-15
496-4
496-7
491-4
½ WATT
PERCENT CHANGE = $\frac{R_1 - R}{R_1}$ 10
½ WATT
496-4
496-7
491-4
0
5
10
15
20
25
30
35
40
45
50
55
60
TIME (MINUTES)

GRAPH A


PERCENT CHANGE VS. TIME
R. BUSCH 4-1-6
RESISTOR
496-R
49?-4
496-7
49I-4
1/2 WATT
R [Ω]
74,000
60,000
11,000
3,950
25,5
1/2 WATT
496-4
498-7
49I-4
0
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
16
17
18
19
20
TIME (HR)

GRAPH B



GRAPH C

R1 = IS RESISTANCE AFTER METALLIZING

R2 = RESISTANCE AFTER HEAT TREATMENT AT 500°F FOR 15 HRS

CONCLUSION

1. The porous quartz substrate is superior to glass substrates used previously. It is better than the porous alumina that has been checked because of better control and therefore, uniformity of the pores.

2. Sensitizing of the substrates is very critical. Metallizing can be accomplished using various methods if sensitizing is done properly.

3. Physical parameters of the substrates could not be related to the resistance value of the substrates. This was due in part to the inability of the fixtures to make good contact on the rough surfaces of the metallized substrates.

4. Of the various terminals tested, a metal frit terminal was selected for use and further study. This terminal requires that the substrates be cut to size and terminated prior to metallizing.

5. Cut substrates can be metallized by use of immersion if the proper sensitizing techniques are observed.

6. A heat treating cycle cannot be used universally to heat treat all resistance ranges. Various cycles will be needed to impart the proper temperature coefficient and stability to a wide range of resistance values.

PROGRAM FOR NEXT QUARTER

1. Determine specific quartz porosities that will be used using air pressure drop and metallizing.

2. Continue experiments with heat treating cycles to match resistor values to the proper cycle for temperature coefficient and stability.

3. Ascertain degree of resistance control obtainable during deposition.

4. Investigate mechanical means of adjustment to value.

5. Continue work on terminations.

6. Investigate protective coatings.

7. Run experiments on wattage ratings.

WESTON INSTRUMENTS
DIVISION OF DAYSTROM, INC.
CONTRACT NObsr 87542
Phase I

February 12 1963

Milestone No.	Milestone Description	1962						1963											
		J	A	S	O	N	D	J	F	M	A	M	J	J	A	S	O	N	D
1	Determination of workable substrate materials *	■	■	■	■	■													
1a	Obtain and investigate ceramic substrate material								■	■	■	▤							
2	Determination of optimum substrate structure *			■	■	■	■	■	■										
3	Determination of optimum chemical solutions for providing uniform film deposition *			■	■	■													
4	Determine method of solution application providing uniform film deposition *			■	■	■	■	■	■										
5	Determination of proper heat treatment								■	■▤	▤	▤							
6	Determination of degree of resistance control obtainable during deposition								■	■	▤	▤							
7	Investigate mechanical means of adjusting to value					■▤	▤	▤	▤	▤	▤	▤							
8	Investigate methods of termination					■	■	■	■	■▤	▤	▤	▤						
9	Investigate spray coatings										▤	▤	▤						
10	Investigate dip coatings										▤	▤	▤						
11	Investigate impregnation and encapsulation										▤	▤	▤	▤	▤				
12	Determine wattage ratings					■	▤	▤	▤	▤	▤	▤							
13	Ship 10 samples of various wattage ratings													▤	▤	▤			
14	Final report														▤	▤			

* Completed