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CRYSTAL CHEMISTRY OF CERAMIC DIELECTRICS

First Quarterly Report

Contract No. DA 36-039 AMC-00107(E)

Continuation of: DA 36-039 SC-78912

Covering Period: 15 November 1962 - 15 February 1963

U. S. Army Electronic Research and

Development Laboratories

Fort Monmouth, N. J.

by

Linden Laboratories, Inc.

State College, Pennsylvania

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This report was prepared by W. R. Buessem and P. A. Marshall, Jr.

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ABSTRACT

The present report consists of three parts. The first part deals with the potential distribution in a BaTiO_3 dielectric under DC-stress and its changes with time. The new measurements which have been performed in dry atmosphere differ markedly from former measurements in humid atmosphere, the other experimental conditions being equal. After degradation had progressed for some time the sample was given rest periods of different lengths after which very strong local fields appeared at the cathode as well as at the anode. It is thought that these fields are connected with the current spikes which had been observed in life tests after similar rest periods.

In the second part of the report the question of whether the proton can enter the titanate lattice is discussed and some new evidence is presented so that it can do so. Reduction of BaTiO_3 in H_2 at intermediate temperatures (500°C) leads to bodies of bright yellow color which is exactly the same color as that of fluorine treated bodies. It is proposed that protons enter the titanate lattice in the form of $(\text{OH})^-$ groups and that the defects which are produced by their entry have energy levels very similar to those of defects formed by F^- ions. The electrical and dielectric properties of H_2 -treated bodies are reported in some detail.

The last part of the report contains a stability study of hot pressed BaTiO_3 dielectrics which we received through the courtesy of the U.S. Army Electronic Research and Development

Laboratories at Fort Monmouth, New Jersey. These samples which have no stabilizing additions like Nb_2O_5 or UO_3 perform remarkably well and reach almost the stability of fluorine treated materials.

II. PURPOSE

This research task shall be comprised of the following three phases:

(Phase a). The development of barium titanate dielectric bodies possessing optimum electrical properties in the temperature range above 300°C. Emphasis shall be placed on exploring the maximum feasible operating temperature and on developing bodies possessing high dielectric strength, low conductivity and electrical dissipating, and long, reliable operating life at elevated temperature.

(Phase b). The investigation of the factors responsible for the electrical conductivity and dissipating in high-K materials other than BaTiO₃ and the development of corrective means to minimize the dielectric losses and increase the useful life of these materials at elevated temperature. Materials to be investigated should include ceramics which are ferroelectric up to temperatures in excess of 300°C, as well as materials which are not ferroelectric in the range from -40 to +300°C.

(Phase c). The investigation of dielectric losses in, and degradation of, moderate-to-low-K high-temperature ceramic dielectrics such as oxides, nitrides, borides and phosphides. This study shall determine the feasibility of a more general application of the methods applied to the degradation of compounds of titanium.

III. EXPERIMENTAL

A. Introduction

A continuation of Voltage Distribution determinations on barium titanate material under new conditions is reported.

The schematic representation for the measurement of the voltage distribution in BaTiO_3 under D.C. stress as a function of time is shown in Figure 2 (see Report 15, 15 November 1962). The measuring device consists essentially of a sample under continuous D.C. stress at elevated temperature, and an electrostatic voltmeter to measure the voltage drop between ground potential and a preselected point on the surface of the dielectric. Care had to be taken to precharge the electrostatic voltmeter to an approximated voltage anticipated at any particular point to reduce the effective charging capacity of the voltmeter ($59\mu\text{f}$). The insulation resistance of the voltmeter is in the order of 10^{15} ohms. The insulation resistance of the system, exclusive of the dielectric, was measured at 200°C to be in the order of 10^{13} ohms. The surface resistance of the sample under measurement should therefore not exceed 10^{11} ohms, in order to obtain reliable voltage readings. For the barium titanate sample reported, the surface resistance at 200°C between a point and ground potential was found to be in the order of 10^{10} to 10^{11} ohms. It will be necessary to increase further the insulation resistance of the system in order to make reliable determinations on dielectric samples with lower surface conductivities. The point probe is positioned with respect to the dielectric by means of a

micrometer screw which allows adjustment to the nearest 0.001 inch. At the present time voltage readings are made at intervals of ten mils along a sample approximately 105 mils total thickness. A microammeter inserted in series with the dielectric permits the reading of total current passing through the dielectric. The entire apparatus is enclosed in a dry box and the ambient water content of the atmosphere is controlled with Drierite (CaSO_4). A discussion of the results obtained by the method is given in Section IV-A.

An experimental study of the effect of the reaction of titanates with hydrogen and water was introduced during this report period. The method of hydrogen treatment was to prepare fired ceramic titanate discs and subsequently refire under controlled conditions in wet and dry hydrogen. An alumina tube furnace was employed in order to confine the atmosphere. A continuous stream of gas was passed over samples at a rate of about 5 liters per hour. Water vapor was introduced by bubbling hydrogen through water prior to delivery to the tube furnace. A split tube furnace was used so that an alumina tube could be inserted conveniently at high temperature. The procedure followed was to preheat the furnace to approximately the temperature desired, while the alumina tube containing the material to be treated was flushed at room temperature. When the furnace reached the desired temperature the gas train was inserted. The time at temperature was measured from the time at which the furnace and gas train came to the desired level. After a given time interval the gas train was removed from

the furnace and allowed to cool to below 200°C before the tube was opened and the samples removed. A discussion of the results are given in Section IV-B.

Life test results are reported on materials hot pressed at the U.S. Army Electronic Research and Development Laboratories. The results are given in Section IV-C.

B. Raw Materials and Sample Preparation

The barium titanate used was obtained from the Titanium Alloy Division of the National Lead Company (Lot 47). The same lot has been used in all studies made thus far involving barium titanate in order to eliminate the variation which would occur from lot to lot. The analysis of the barium titanate as reported by the manufacturers is shown in Report No. 12, 15 October 1961, Table I.

As a general procedure additions of oxides were accomplished by the addition of nitrate solutions or ammonium salt solutions. Additions made in this manner result, upon decomposition, in a finely divided, highly reactive form which enhances chemical reaction. In those systems where the appropriate soluble salts were not available, the oxide additions were wet-milled into the base dielectric raw material, i.e., BaTiO_3 , TiO_2 , CaTiO_3 , NiO .

Bodies were formed by pressing the dried powder in a steel mold to 6000 psi.

C. Firing (General Procedure)

Raw samples were placed on refractory slabs which were covered with zirconia powder to avoid contamination by reaction with the refractory slab. The bodies thus prepared were fired in a continuous fashion in an electric globar tunnel kiln. The maximum firing temperature for BaTiO_3 was 1450°C and the firing cycle was such that the samples were subjected to this temperature for approximately one hour.

Electrodes of silver, gold, graphite or indium-gallium alloy were applied to the samples, depending upon the use for which the same was intended. The silver was a duPont colloidal silver preparation (No. 5326) which was fired to a well-adhering electrode at 700°C for one hour. A colloidal suspension of silver in a volatile vehicle but not containing glass as a binder was used, employed for low temperature electrode application (courtesy of Handy and Harman, Bridgeport, Conn.). The gold preparation was obtained from the L. Reusche and Company under the name of Liquid Bright Gold No. 31. Samples painted with the gold complex were fired to 700°C for one hour in order to follow the same heat treatment as in the case of application of silver electrodes.

Gold electrodes were used on all samples which were to be subjected to life-test determinations. The problem of "Silver creep" was thus eliminated as a factor contributing to degradation.

Graphite electrode material was Grade CS obtained from the National Carbon Company.

IV. RESULTS AND DISCUSSION OF RESULTS

A. Voltage Distribution under DC-Stress as Function of Time

The first experiments on voltage distributions and their changes with time under life-test conditions (200°C, 9.35 volt/mil) were reported in Report No. 14 (15 April 1962); the samples there consisted of commercial grade BaTiO₃ without additions, in the shape of cylinders 108 mil in diameter and 108 mil high. These measurements were followed up by voltage distribution determinations on the same material under identical conditions but with a sample diameter of 270 mils (see Report No. 15, 15 July 1962).

In addition to the two sizes (108 and 270 mil diam.) samples of two larger sizes (nominal diameters 1/2" and 1") were prepared from the same lot of BaTiO₃. However, it was found that these two larger size samples could not be tested under the same conditions because they deteriorated too fast, e.g., the 1" diameter sample broke down within two minutes. The comparison of the four sizes 108, 270, 500 and 1000 mil diameter, in which the degradation speed rapidly increased with the diameter under the same voltage stress conditions, forces one to the conclusion that thermal effects play a dominant role in the degradation of this material at 200°C under 9.35 volt/mil stress, at least for all sizes greater than 108 mil diameter. This should not be so if the heat transfer were mainly effected by the end faces (electrodes), since the end-face area increases exactly in proportion to the volume

under electrical stress, i.e., to the nominal power input. It follows then that the cylindrical side faces which increase only linearly in area with the increasing diameter must play an important role in cooling the samples.

In an attempt to investigate some other variables and their effect on degradation, the experiments of Report No. 15 (270 mil diam. samples) were repeated with several modifications. First, the time was extended from 4 hours to 70 hours. Secondly, special care was taken to keep the atmosphere around the sample very dry; the humidity was less than 5%. This compares with about 60% humidity in the earlier experiments. Thirdly, three rest periods were scheduled during the later stages of the degradation process. This was done to bring about some "spikes", i.e., the large current increases after rest periods which were studied in the last report. It was hoped that more would be discovered about the field and space charge conditions responsible for this phenomenon.

The results of this new series are presented in Figures 1-22. Figures 1-5 can be compared with the corresponding Figures 27-33 of Report No. 15 (15 July 1962) which give the field distribution of the same samples measured at corresponding times under humid conditions.

It can be seen that the two series are not identical. Whereas in the earlier series a positive space charge developed in the front of the anode and extended far towards the cathode with a narrow negative space charge developing at the cathode after 25 minutes, the new series showed a negative space

charge (s.c.) in front of cathode and anode right from the start and a rather constant small positive s.c. (2 Coul/m^3) in the interior for the first five hours. During this time, the negative s.c. in front of the anode increased to very high values (375 Coul/m^3) and led to a voltage drop of 26000 v/cm . Between 5 and 23 hours the negative s.c. at the cathode disappeared and the positive s.c. in the interior contracted to a narrow band of 20 mil which after 29 hours reached the cathode, intensified and reached peak values of 37 Coul/m^3 . At this time the negative space charge at the anode had increased to 550 Coul/m^3 .

Since degradation had now progressed considerably, a rest period of 37 minutes was interposed. After reapplying the field the space charge in front of anode and cathode both increased strongly and the local fields there reached values of 44000 v/cm (Fig. 12). After 2-1/2 hours the space charge at the cathode had decreased whereas the field at the anode was still increasing.

A second rest period of one hour (Fig. 16) again caused an increase in the space charge at cathode and anode. The local field at the anode now reached 48000 v/cm which was 15 times the average applied field. The space charge at both cathode and anode decreased during the next hour.

A third rest period of 16 hours led to new strong increases in the fields at both cathode and anode. The cathode field was now 52000 v/cm ; after two hours the field values dropped to about 20000 v/cm at the cathode and 32000 v/cm

at the anode. After another 75 hours (Fig. 21) these high values were still almost unchanged.

By dividing the local field by the total current and multiplying this value by the area, an apparent local resistivity can be computed; these values are given in Figures 21 and 22 for seven positions inside the sample as a function of time.

At the same time that Report No. 15 was printed, an important paper by Lehovc and Shirn⁽¹⁾ was published. Since this paper is highly relevant, it shall be discussed in some detail. In Table I, the experimental conditions for both investigations are compared.

TABLE I

Comparison of Experimental Conditions on Potential Distribution Experiments by Lehovc and Shirn⁽¹⁾ and Linden Laboratories (Reports No. 14, 15, 17)

	<u>Lehovc and Shirn</u>	<u>Linden Labs.</u>
Material:	Commercial High K body (BaTiO ₃ with CaTiO ₃ etc.)	Commercial grade BaTiO ₃ (no additions)
Curie Temp.:	35°C	123°C
Field:	6, 4 and 2 KV/cm	3.75 KV/cm
Temp.:	250°C, 300°C	200°C
Thickness:	2500 and 5000 micron	2720 micron
Area:	4x10 ⁻³ cm ²	5.8x10 ⁻² and 36.3x10 ⁻² cm ²
Time:	63 hours	43 and 71 hours
Electrodes:	Aquadag (Graphite)	Gold
Instr.:	Electrostatic Voltmeter	Electrostatic Voltmeter

The main differences between the two series of experiments are in the material, in the temperature, in the area and in the electrode material.

The main difference in the results of L. & S. is the see-saw effect in most of their current vs time curves consisting first in a strong rise in the current, then an even stronger decrease and then another rise. This we did not find, i.e., our curves resemble more the curve III in L. & S.'s Figure 1 and not their curves I and II, Figure 1, or their curve I in Figure 2.

As it happens, practically all of the subsequent evaluations in L. & S.'s paper deal with the phases in the current history of their samples in which the current decreases by one or more orders of magnitude. On the other hand, all of our potential distribution curves and subsequent evaluations deal with the phase in the current history of our samples in which the current increases by one or more orders of magnitude. It is, therefore, not surprising that L. & S.'s potential distribution curves and all their derivatives are quite different from ours. That is necessarily so because the current decrease or increase must be in a causal relation to field distribution inside the dielectric and, therefore, the distributions for a phase of strongly decreasing currents cannot be the same as that for a phase of strongly increasing currents.

The main difference in the space charge distributions in the two cases seems to be that in the case of increasing currents strong space charges are built up directly in front

of the anode (Fig. 4) and somewhat later in front of the cathode (Fig. 8), whereas in L. & S.'s experiments during the decreasing current phase the main space charges are in the interior where they move from the anode towards the cathode. From this it might be tentatively concluded that the strong increase in current is connected with strong rising fields at the anode as well as at the cathode. This is also born out by the behavior of sample I in Figure 2 in L. & S.'s paper during the first 132 minutes, which is a period of increasing current. Summarizing the discussion of L. & S.'s paper it can be said that for some reason the second period in L. & S.'s current vs time pattern, which is called "conductivity extraction" or "resistivity injection" at the anode, is missing under our conditions.

It is impossible to say which of the different conditions in Table I: material, field strength, temperature, electrode area, electrode material, is responsible for this fact. Unfortunately, it is not possible to change all these variables independently, since for a given body composition only a narrow field strength/temperature combination can be used. Our BaTiO_3 material cannot be subjected to the conditions employed in L. & S.'s paper because it would break down in seconds, nor can L. & S.'s more stable material be investigated under our conditions because it would only show very slow changes; furthermore, the time constant of the electrostatic voltmeter would be prohibitively long.

In the light of the results of this report, two variables which can be varied independently seem to be of sufficient

importance to warrant a more systematic investigation: humidity and electrode material; it is hoped that studies in this direction will give further clues as to the physical nature of the degradation process.

B. Effect of Hydrogen and Water

1. Introduction

The difference in potential distribution under DC-stress under dry-atmosphere and humid-atmosphere conditions, which has been shown in the preceding chapter, focuses the attention again on the role of the proton in titanates. Since the beginning of this work some speculation has been done as to the possible effect of water present in the atmosphere and at the surface of the specimen during testing on the degradation of titanates. The possibility that protons could have some effect and even could play a very important role in causing degradation damage has been stressed already in our very first report.

Besides the observations discussed in the preceding chapter, other facts contributed to the renewed interest in the proton and its possible effects on titanates.

There are, first, observations on the strong effect of water vapor during certain stages of the sintering process of BaTiO_3 , on the density and other properties of BaTiO_3 ⁽²⁾.

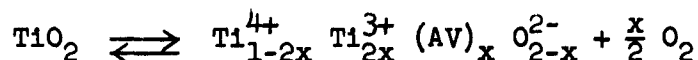
Secondly, there is the increasing importance of the reduction of BaTiO_3 at intermediate temperatures in the manufacturing process of barrier-layer capacitors of the hypercon, etc.-type (trade name).

The question then arises as to what are the reactions of solid titanates with H_2O , H_2 , O_2 and (OH) at any temperature, and what are the reaction products. What is the effect of such reactions on the stability of titanates under lifetime conditions? The original question of whether protons might not enter the titanate lattice during life test and contribute to degradation has now to be extended to the more general question of whether the protons enter the titanate lattice at any time, during firing, reduction, silver firing and life test and, if they do, in which way they effect the properties of interest, especially lifetime.

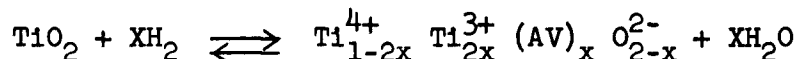
The role of H_2O , O_2 , H_2 and (OH) in titanate reactions is discussed in the following, and strong arguments are brought forward that the proton can be built into the titanate lattice. The proton is present in the form of $(OH)^-$ groups and the complete analogy of these $(OH)^-$ groups to the $(F)^-$ groups, which were discussed in great detail in the last report, is one of the most striking features of our findings.

2. Observations on Polycrystalline Titanates under Mild to Severe Reducing Conditions

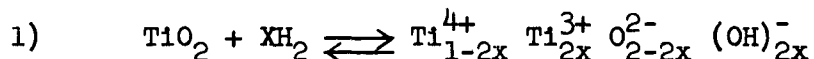
It is well known that rutile or barium titanate becomes a black semiconductor under conditions of strong reduction. This has been attributed to the loss of O^{2-} ions on sites within the crystal lattice and the generation of an appropriate number of Ti^{3+} to maintain electrical neutrality in the smallest possible volume element. The chemical equilibrium could be described as follows for a low oxygen-pressure system,



or in hydrogen as,



However, it has been demonstrated⁽³⁾ that reaction of hydrogen with single crystals of rutile at intermediate temperature, 800°C, led to the formation of OH⁻ groups within the crystal supporting the fact that hydrogen migrates into the lattice. The presence of OH⁻ groups was confirmed by infrared spectra. Von Hippel⁽³⁾ further reported that a saturation of OH⁻ groups is reached and suggests that a competing reaction may begin which interferes with further OH⁻ formation. The above can be described chemically as follows:



The reaction of TiO₂ with hydrogen leads to a forced reduction of some of the Ti⁴⁺ to Ti³⁺ to maintain electrical neutrality; however, as the concentration of OH⁻ reaches a critical value, water can condense out with the result being the formation of an anion vacancy. Von Hippel summarizes his findings in the following way: "... even when only spurious amounts of hydrogen or water vapor are present the OH bands appear rapidly at elevated temperatures, i.e., rutile seems to be an excellent hydrogen detector and scavenger."

During this report period similar experiments involving reactions of polycrystalline barium titanate with H_2 and water vapor were undertaken. Reductions in dry hydrogen were carried out at 500° , 600° , 700° , 800° , 900° and $1000^\circ C$, for 10 minutes and a second series for 30 minutes. It was found that dry H_2 was, as expected, the most efficient reducing agent. Hydrogen which was saturated with water vapor was not an effective reducing agent. Table II gives the visual results using color as an index of the degree of reduction.

It should be noted that at $500^\circ C$ the reaction of H_2 with barium titanate led to a yellow color. For purposes of comparison this is the identical color which was obtained by the introduction of fluorine into the barium titanate crystal lattice. Indeed, the above color sequence going from yellow to black can be mimicked by fluorination.

The similarity between F^- and OH^- might be considered the justification for the analogous visual appearance. At this point however the similarity must cease since an OH^- is best considered as an O^{2-} plus a H^+ and it is quite probable that the proton can migrate in a field and thus contribute to the mechanism of degradation under a direct current stress. This may be suggested as a possible mechanism to account for the discoloration which occurs under the cathode of a titanate dielectric after a given stress history.

TABLE II

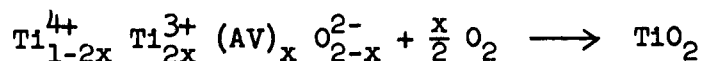
BaTiO₃ Reduced in Hydrogen

<u>Sample No.</u>	<u>Composition</u>	<u>Reduction Temp. °C</u>	<u>Time (Minutes)</u>	<u>Color, Remarks</u>
	BaTiO ₃	---	--	Tan
H-41	"	500	30	Yellow
H-42	"	600	30	Yellow-green
H-43	"	700	30	Yellow-gray
H-44	"	800	30	Gray-black
H-40	"	900	30	Black
H-39	"	1000	30	Black
H-22	"	1000	10	Black
H-27	"	1000	10	Yellow-gray (in wet hydrogen)

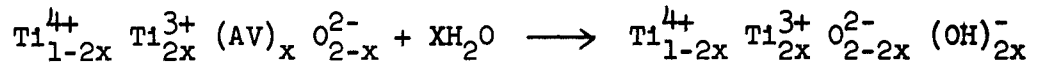
It has been demonstrated that blue spikes surrounded by a yellow periphery can be found under the cathode after a given stress history. [(First Quarterly Report 1 July 1956 Contract No. DA-36-039-SC-71190. Study of the Degradation of High K Ceramic Dielectrics)]. Von Hippel's findings show that OH⁻ groups in single crystalline rutile can only reach a given saturation (concentration) before a secondary reaction occurs to limit saturation and suggest that water vapor is the product of condensation. A similar proposal can be made on polycrystalline titanates under stress where the saturation limit is reached as a result of migration of H⁺ in the direction of the cathode. As the concentration is increased the probability of condensation of two OH⁻ groups to form water and an anion vacancy is increased; therefore, in the immediate vicinity of the cathode the material would be blue-black, whereas the boundary between the blue and the tan color of the bulk would be the color of the titanate with a high concentration of OH⁻. From Table II the most probable color for this boundary layer would be yellow.

The omnipresence of water vapor in the atmosphere justifies the thought that barium titanate would contain OH⁻ groups under what is referred to as normal firing conditions.

At high temperatures the titanate can be considered to be strongly deficient in oxygen and upon cooling is re-oxidized by reaction with the atmosphere as follows:



However, reaction with water vapor must also be considered to be equally probable:



The degree of reaction is dependent on the water vapor pressure. Therefore, a "normal blank" upon removal from a furnace must be considered to be a summed effect of the two above reactions as the temperature is lowered. The latter reaction could conveniently account for at least some of the difficulties encountered in attempts to obtain duplicate electrical properties from materials fired from the same batch preparation but at different times.

For the same reason, water vapor interferes with the reduction of barium titanate in a hydrogen atmosphere. Therefore the dew point of a furnace atmosphere should be monitored whether the purpose of the firing is for the preparation of normal dielectrics or for the purpose of producing a reduced body as an intermediate step by making a "barrier layer" dielectric.

Further work on this phase of the program will be conducted to demonstrate the influence of the water vapor pressure of the furnace atmosphere on the electrical characteristics of the firing of dielectric bodies of identical composition.

3. Effect of Hydrogen Treatment of Commercial Body on Life Time

A commercial K 6000 body was subjected to a hydrogen atmosphere at 500°C for 10 minutes. Six such samples, along

with four K6000 blanks (no treatment by Linden) were placed in life test at 200°C and 20 V/mil.

Prior to placing the above in life test it was necessary to determine the conditions (temperature and stress) at which a reasonable life expectancy could be had from the unadulterated bodies (K 6000 blanks). For this purpose the temperature was maintained at 200°C and the stress changed from 30 to 25 to 20 V/mil. The following is a summary of the results at those three stresses:

	<u>Lifetime</u>
30 V/mil Temp. 202°C	81 min.
25 V/mil Temp. 201°C	172 min.
20 V/mil Temp. 200°C	>4320 min.

The life test was performed at 20 V/mil; six of the test samples were treated in H₂ at 500°C for 10 minutes, four control samples without H₂ treatment were also tested. The results are shown in Figures 23 and 24. The curves representing the untreated body warrant some comment, especially since one sample (#7) failed after seven hours, which was not expected from the above test results.

After a current surge between 2-5 hours the current increased on all samples to between 400 and 1000 μ a. and varied considerably; no fuses blew (32ma) until 6.66 hour when sample #7 failed. Samples 4, 5 and 8 began decreasing the current drain and at eight hours were drawing 350, 303 and 340 μ a respectively.

After 24 hours the current dropped to 13.3, 10.5 and 8.5 μ a respectively. The current continued to decrease, and after 172 hours, the run was discontinued.

The effect of the 500°C H₂-treatment on the body is, on the whole, unfavorable. The beginning of the great current surge is shifted from 2-3 hours to 5-7 hours but all six samples failed between 4 and 8-1/2 hours.

4. Effect of H₂-Treatment on Conductivity, Dielectric Constant and Tan Delta of BaTiO₃

The BaTiO₃ samples which were characterized as to their color after H₂-treatment (IV-B-2) were subjected to an extensive investigation of their conductivity, dielectric constant and loss factor. The purpose of these measurements was to find some evidence for reaction 1) (IV-B-2), i.e., for the presence of (OH)⁻ groups in the lattice and their effect on the electric properties.

The results of these measurements are presented in Figures 25-35 (IV-B-4).

Since both reactions 1) and 2) result in conductivity (Ti³⁺), it is too early to discuss these measurements at this stage because all that can be recognized clearly on these samples can be described by conductivity effects. It is hoped that later investigations by infrared techniques will allow the effects due to the pair Ti³⁺/(OH)⁻ to be separated from those due to the trio 2 Ti³⁺/(AV)²⁺.

C. Life Test of Hot-Pressed BaTiO₃ Samples

The test results on three hot-pressed BaTiO₃ samples, prepared by the U. S. Army Electronic Research and Development Laboratories*, Fort Monmouth, New Jersey, are presented in Table III and Figure 36.

The stability of these samples (which contain no stabilizing agent such as U⁶⁺, Nb⁵⁺ or F⁻) is outstanding. In the light of our ideas on stability, this is due to the purity, density and, especially, the homogeneity of these materials.

For comparison, it might be mentioned that similar stabilities could be achieved with commercial bodies only by fluorine treatment (see Report No. 16, 15 December 1962, p. 19, line 13).

* Test pieces furnished by Mr. R. Brandmayr

TABLE III

Current in μ amp

Hot-Pressed Samples from USAF/RD Laboratories
 200°C, 80 mils Thick, 30 V/mil, Gold Electrodes to Edge

	Time	Temp.	Sample #2	Sample #3	Sample #9	
Feb. 5	0934	199.0	0+	0+	0+	
	1740	200	0	0	0	
Feb. 6	0917	198.7	0	0	0	
	1600	199	0	0	0	
Feb. 12	0907	201	0	0	0	
	1300	202	0	0	0	shut down for rest
	1417	201.5	0+	0+	0+	
	1428	201.5	0	0	0	
	1700	203.8	0	0	0	shut down for rest
Feb. 14			power was off from Feb. 12, 1700 hrs., increased temp. to 250°C before reapplying power			
	1504	249	1.0	.8	.3	
	1505		.5	.3	.1	
	1512	250.5	.2	.1	.1	
	1753	251.5	.1	.1	.05	
	0940	248	.1	.1	.1	
	1230	250.2	.2	.2	.2	
	1000	246	.05	.05	.05	
	1015	244	.05	.05	.05	
	1030	260	.05	.05	.05	discontinued; raised temp. to 300°C
Feb. 15	1300	299	4.5	2.0	1.5	
	1301.5	299	.9	1.7	1.1	
	1307	298	.6	1.3	1.0	*visible arcing occurred on electrode surfaces of these samples, test discontinued
	1800	306	.5	1.8	1.0	
Feb. 21	1030	294	.9	1.7	2.5	
	1645	298	1.0	2.0	3.8	
Feb. 22 *	1227	294	1.8	1-1.5	1.5-2.5	

REFERENCES

1. K. Lehovec and G. A. Shirn, Conductivity Injection and Extraction in Polycrystalline Barium Titanate, Journ. Appl. Physics, 1962 (June) 33, 2036-2044.
2. E.g., U.S.A. Patent #2,696,651 (Clevite) 1955
U.S.A. Patent #3,049,431 (Gen. Electric) 1962
See also the property sheets of BaTiO₃ lots, manufactured by Tamco, which show consistently a greater density when fired in steam as compared with densities obtained in standard air firing.
3. A. v. Hippel et al. MIT Lab. for Insulation Research, Contracts AF 19 (604) - 6155 and Nonr 1841 (10).
Report June 1961.

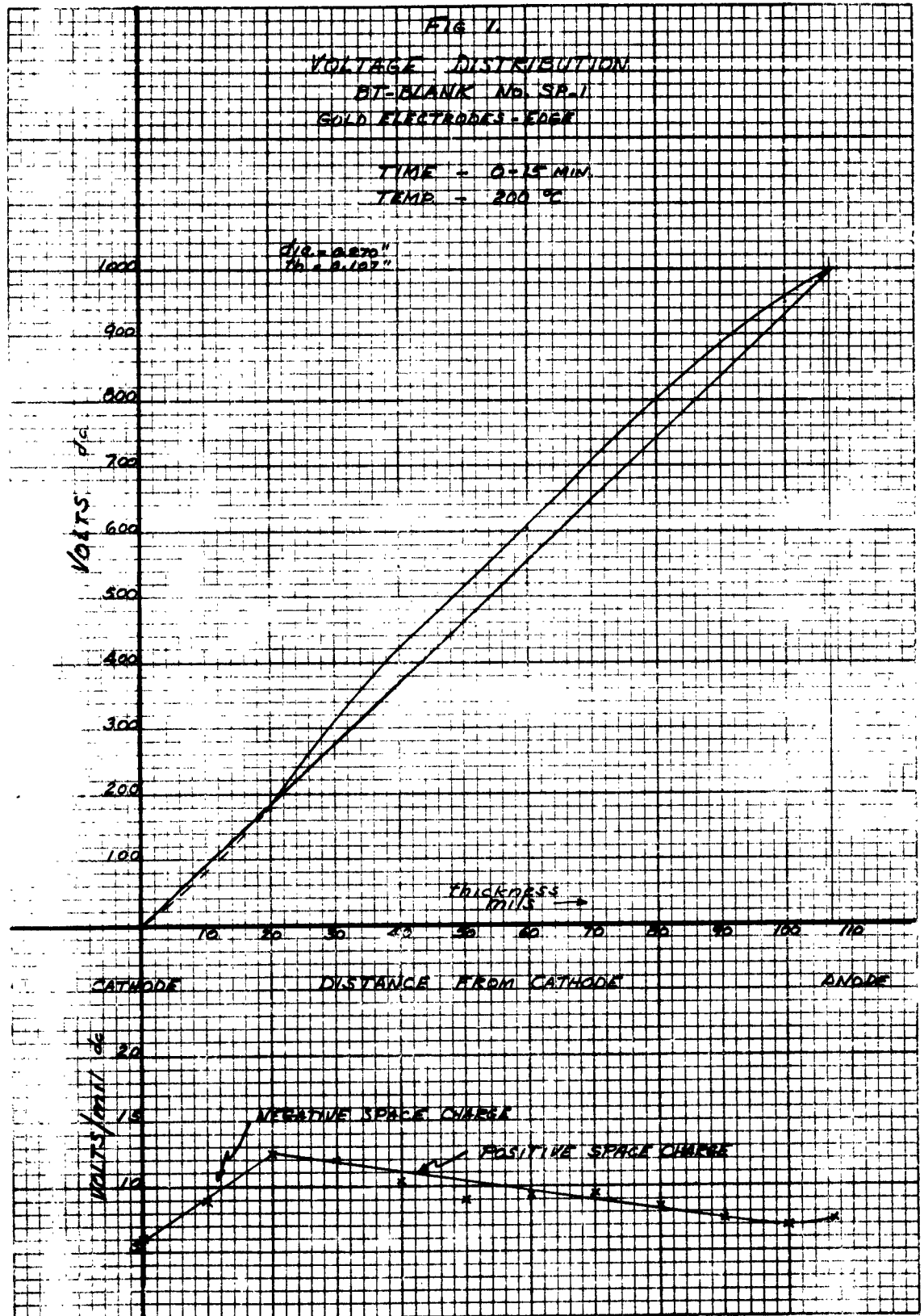


FIG 2
VOLTAGE DISTRIBUTION
AT-BLANK NO. 5A.1 (CONT.)

TIME - 30-45 MIN.
TEMP - 200 °C

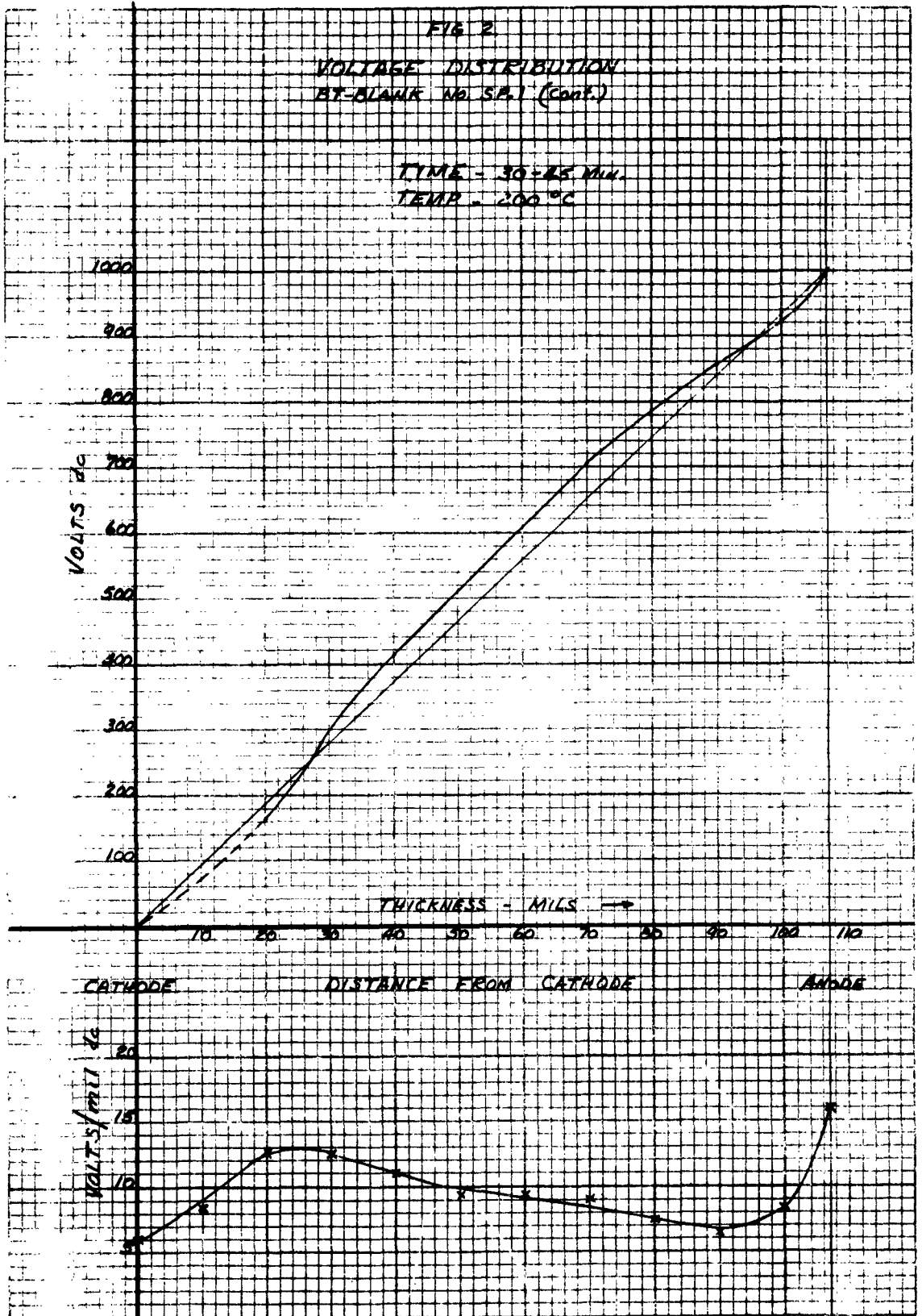


FIG 3
VOLTAGE DISTRIBUTION
BT-BLANK No. SP-1 (cont)

TIME - 1.25 - 1.5 HRS
TEMP - 200 °C

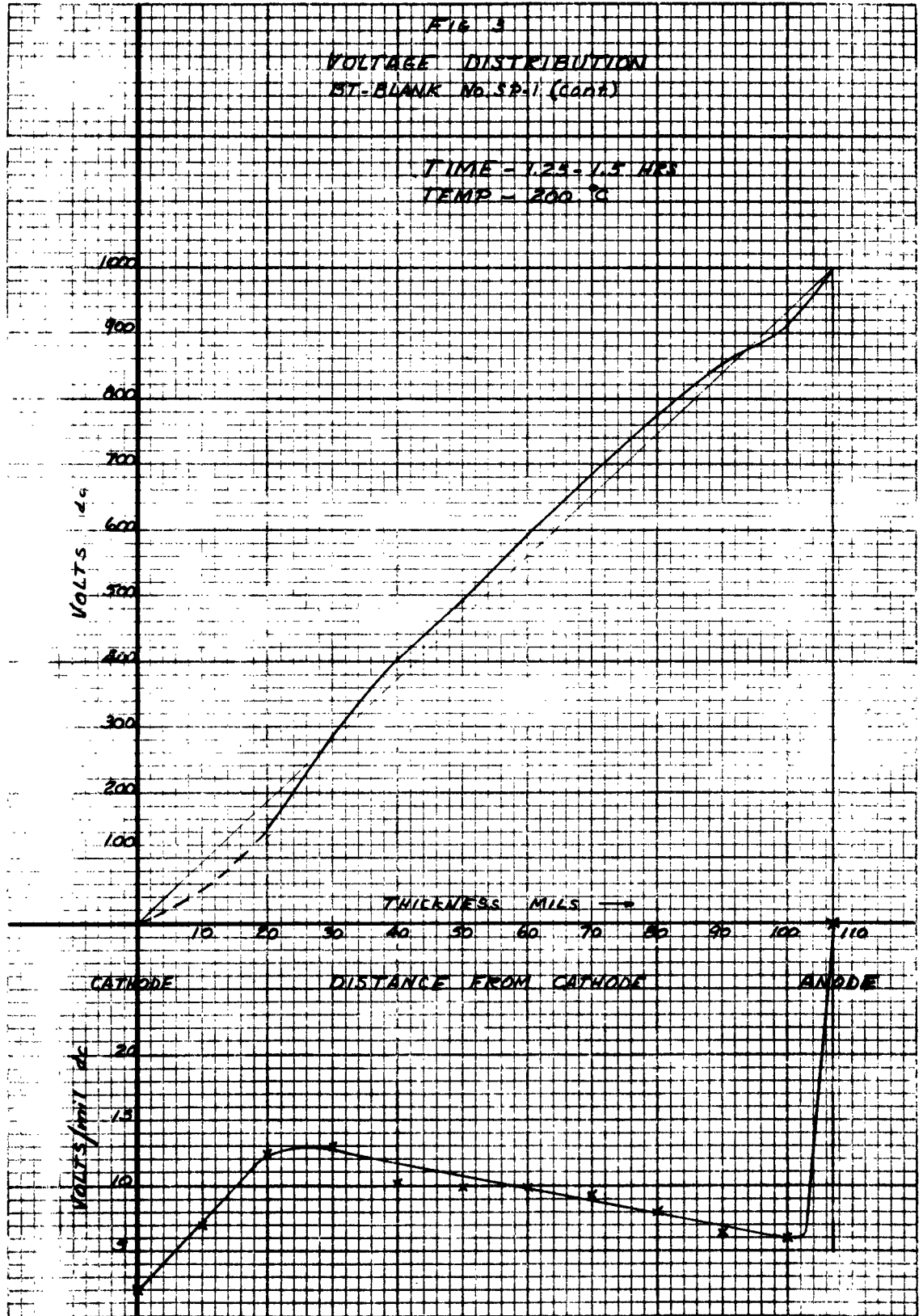


FIG. 4

VOLTAGE DISTRIBUTION
BT-BLANK No. SP. 1 (Cont.)

TIME - 3.0 - 3.25 HRS
TEMPER - 200 °C

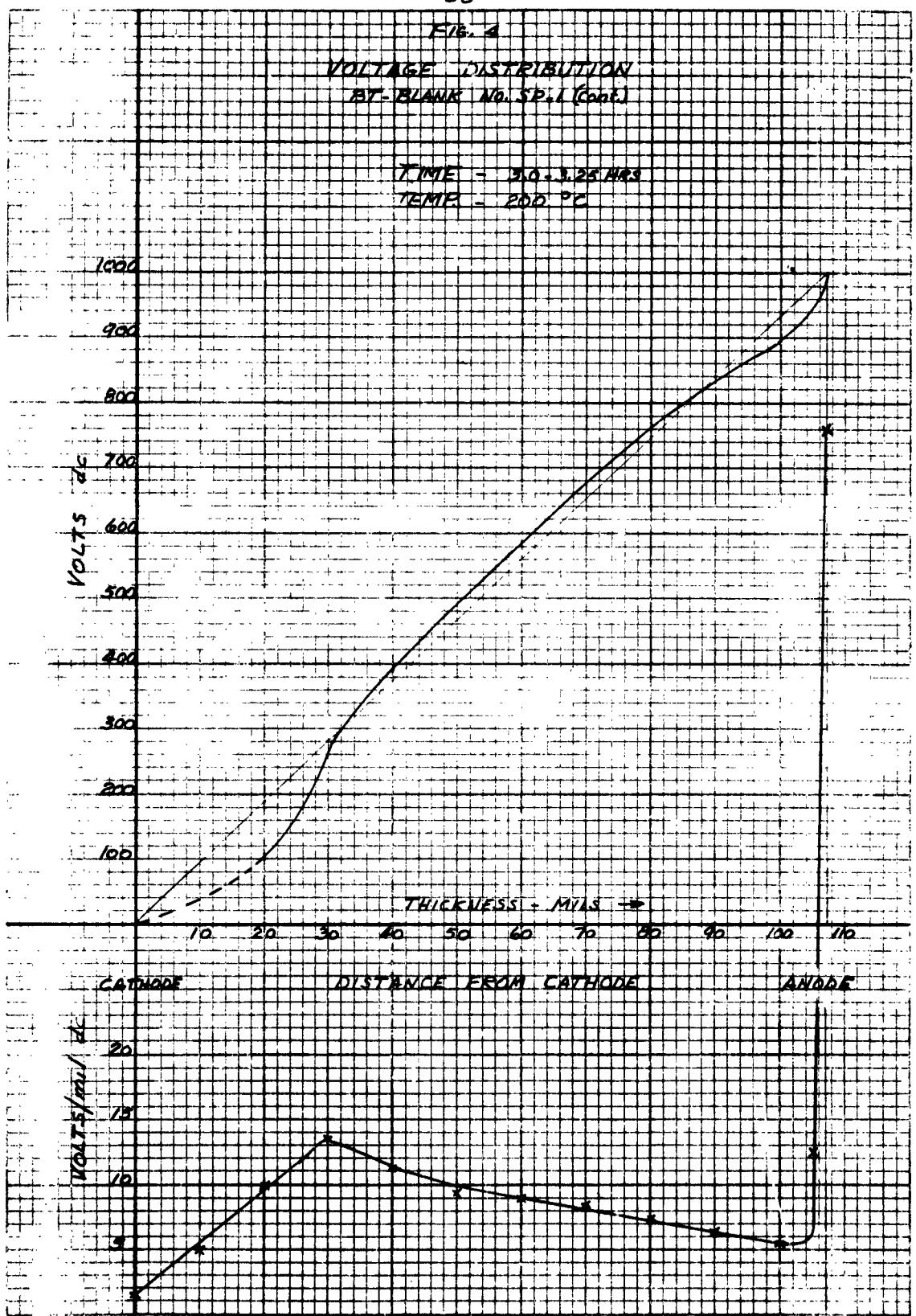


FIG. 3

VOLTAGE DISTRIBUTION
BY-BLANK NO. SP. 1 (CONT.)

TIME - 5.0 - 5.25 HRS.
TEMP - 200 °C

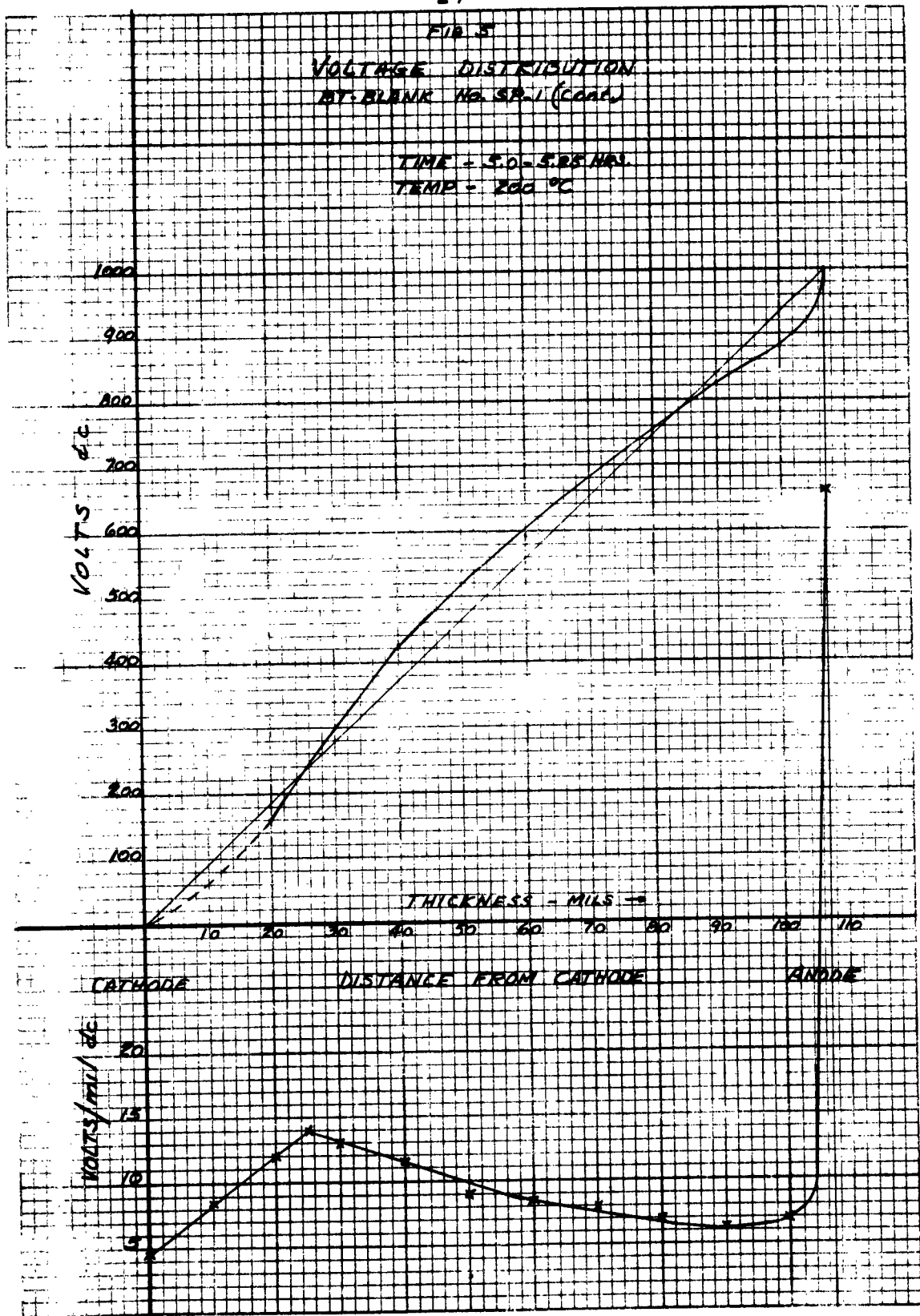


FIG 5

VOLTAGE DISTRIBUTION
BT. BLANK NO. SP. 1 (E005)

TIME - 23.5 hrs

TEMP - 200 °C

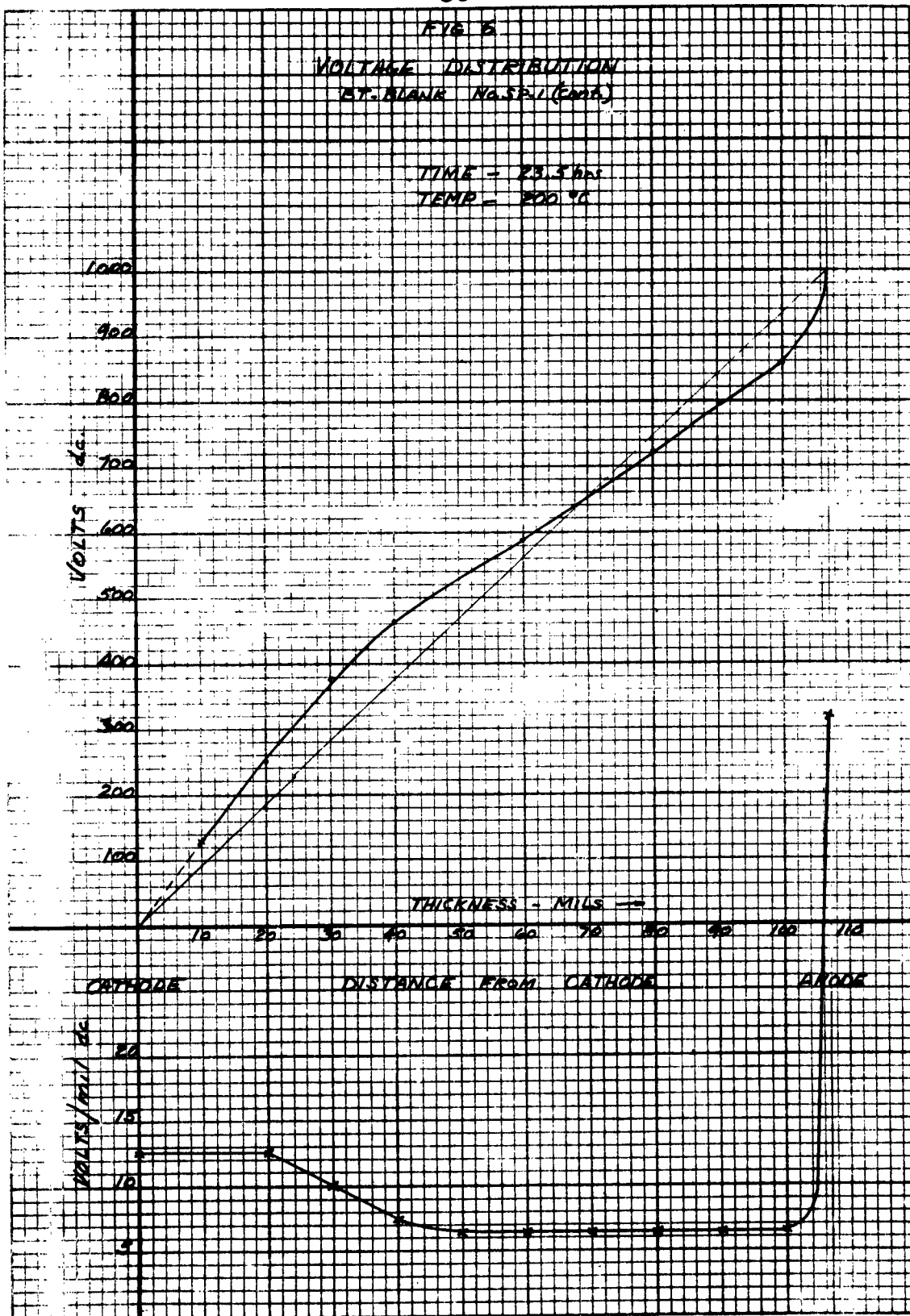


FIG 7
VOLTAGE DISTRIBUTION
BT-BLANK No. SP-1 (cont.)

TIME - 27.5 MRS
TEMP - 200 °C

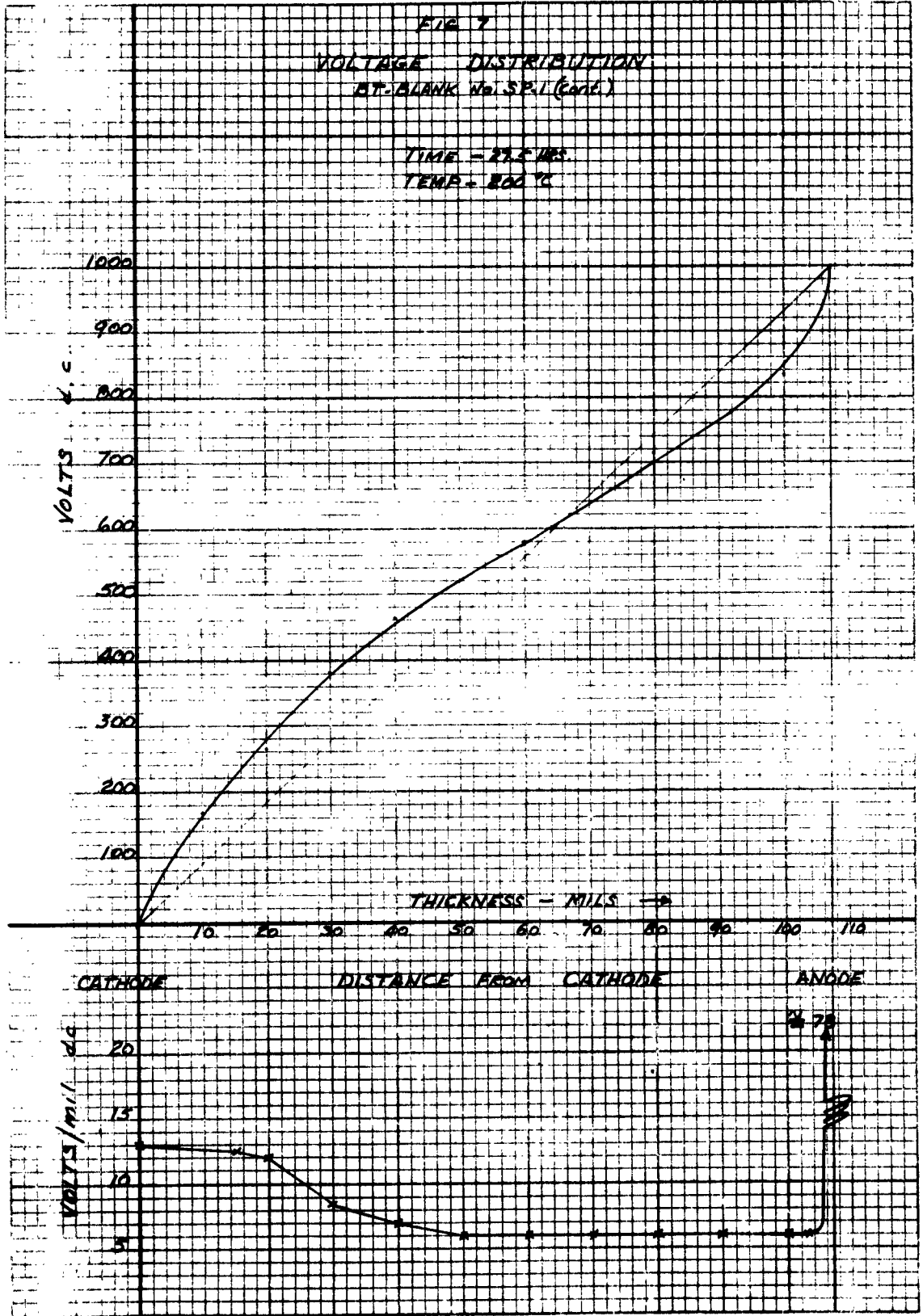


FIG. 8
VOLTAGE DISTRIBUTION
BT-BLANK NO. 56.1 (Genb.)

TIME - 24 HRS.
TEMP. - 200°C

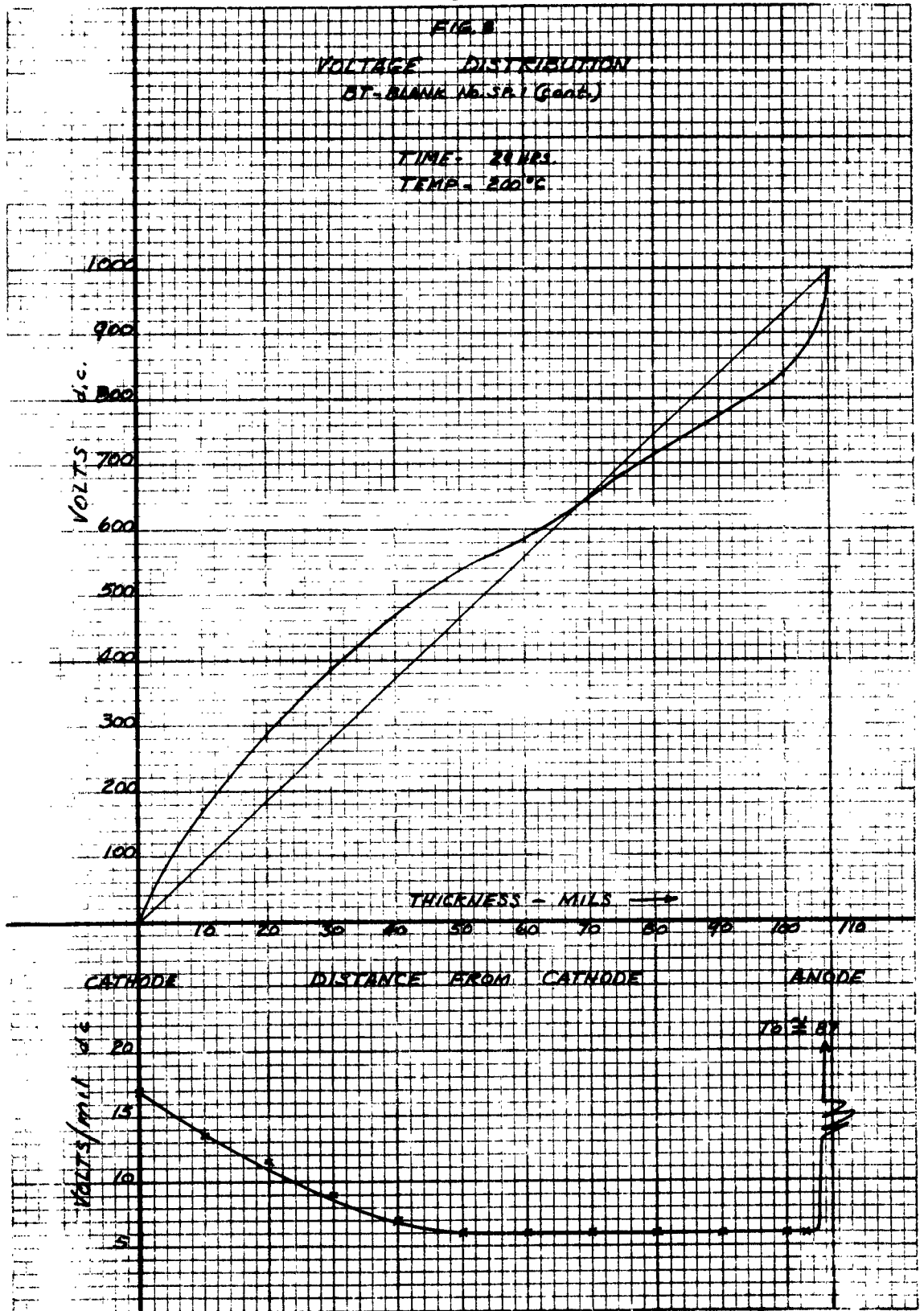


FIG 9.

VOLTAGE DISTRIBUTION
BT-BLANK No. SP. 1 (CONT.)

TIME - 45.5 HR.
TEMP - 200 °C

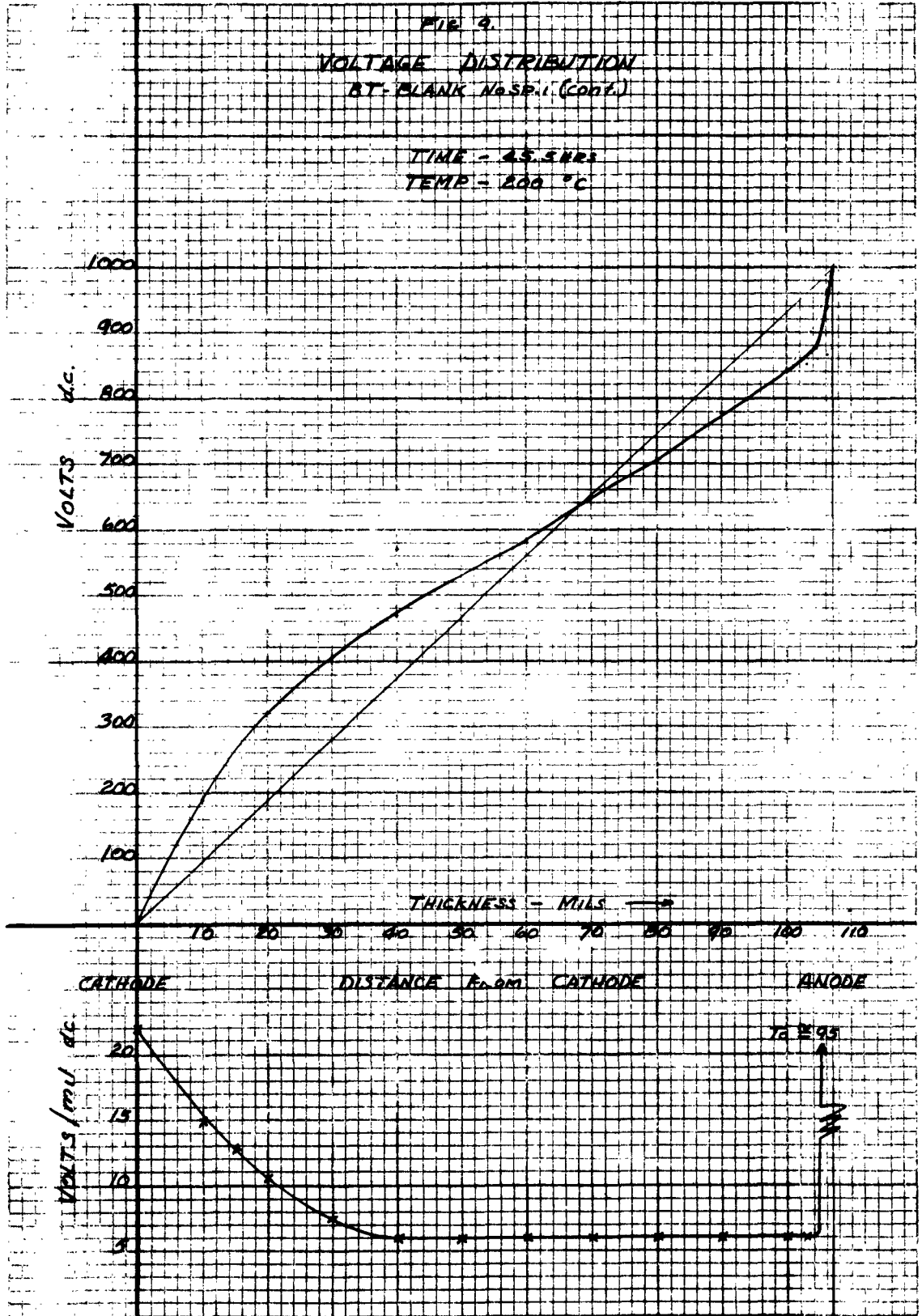


FIG 19
VOLTAGE DISTRIBUTION
BT-BLANK NO. SP-1 (CONT.)

TIME - 50.5 MRS.
TEMP - 200 °C

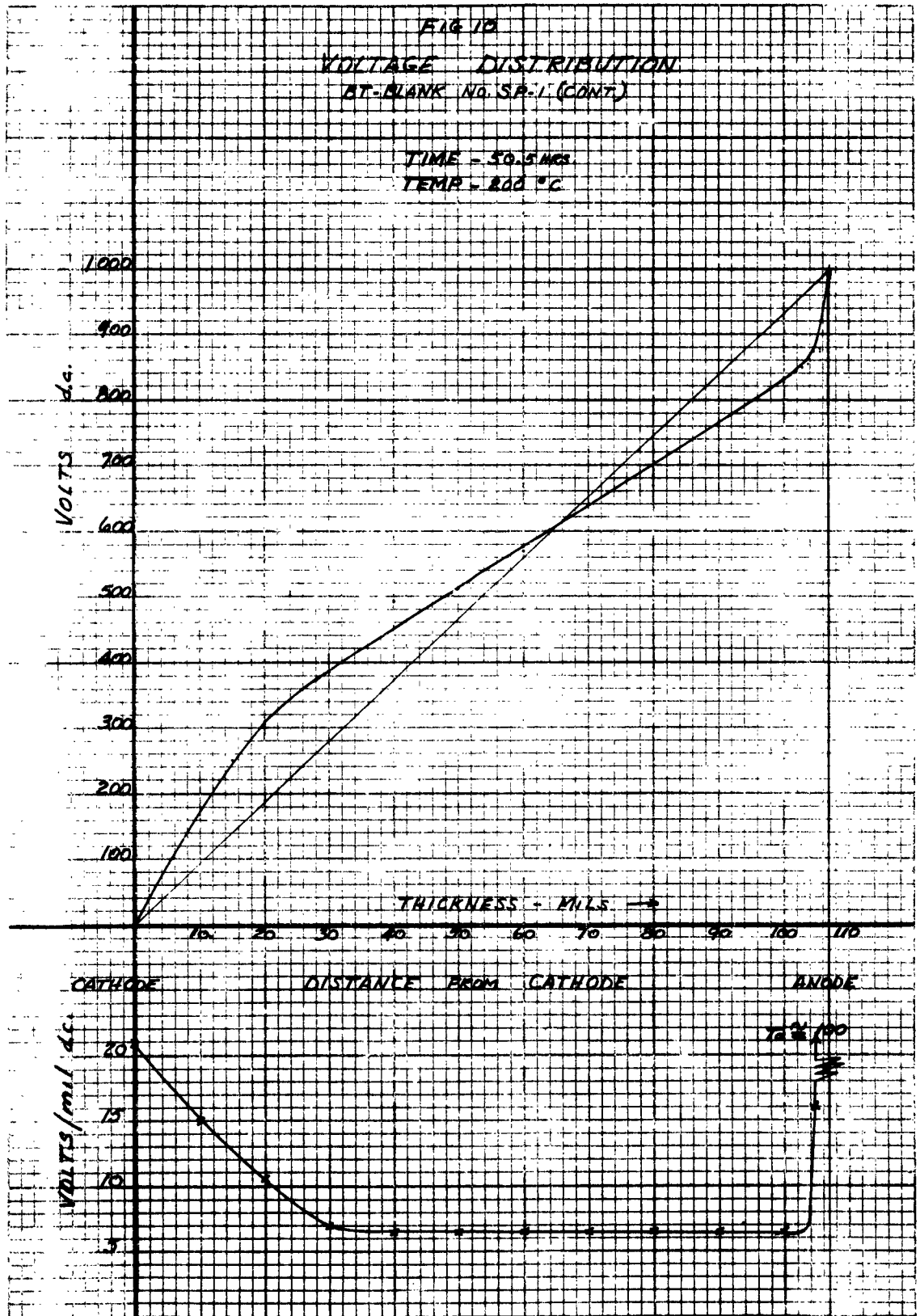


FIG. 11
VOLTAGE DISTRIBUTION
AT BLANK NO. SP-1 (CONT.)

TIME - 70.6 HRS
TEMP - 200 °C

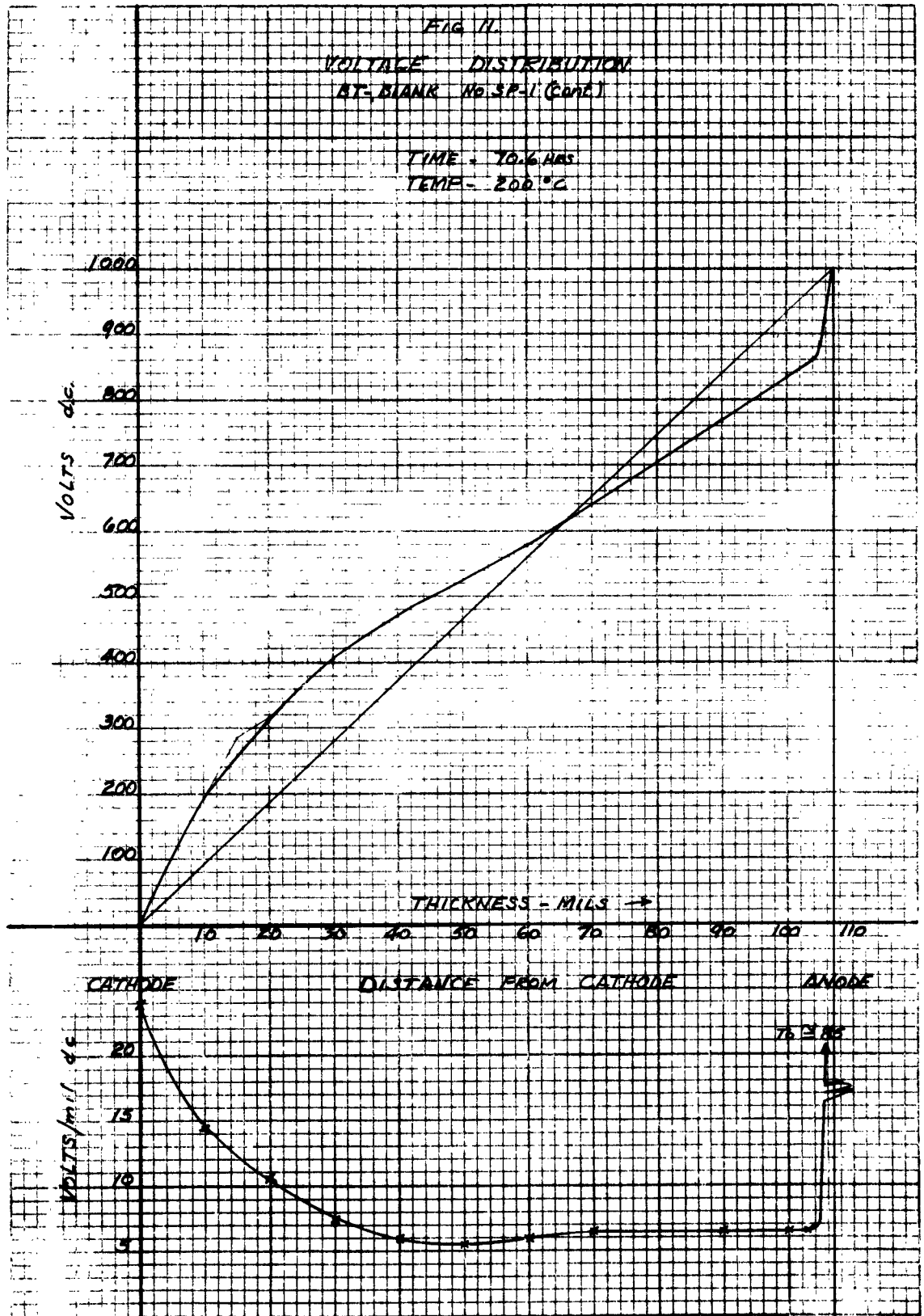


FIG 12
VOLTAGE DISTRIBUTION
BT-BLANK No. 3P1 (cont.)
AFTER 37 min REST
TIME - 0-10 min
TEMP - 200 °C

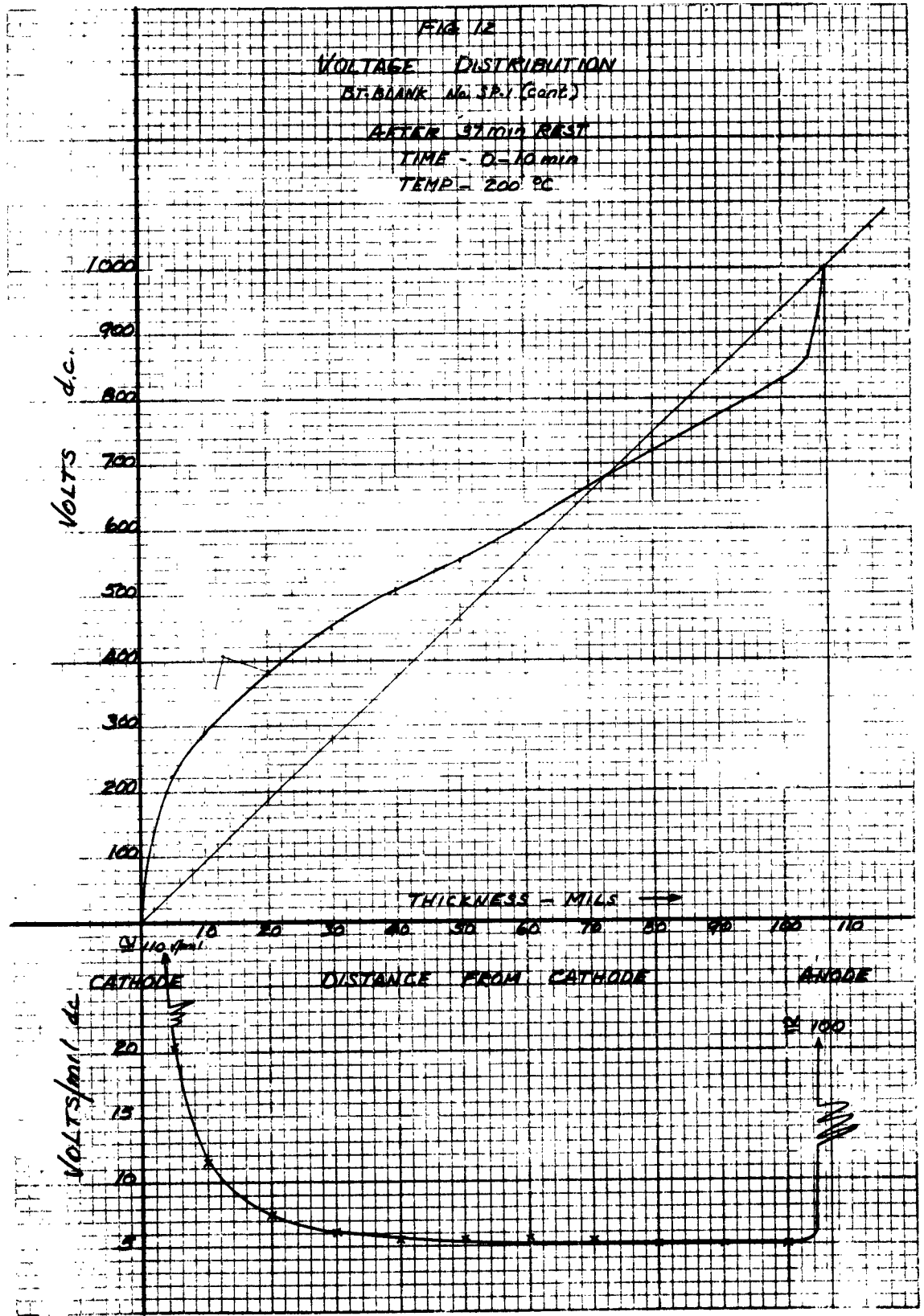


FIG 13
VOLTAGE DISTRIBUTION
AT BLANK NO. 381 (CONT.)
METER 37010 RST
TIME - 18-31 min
TEMP - 200 °C

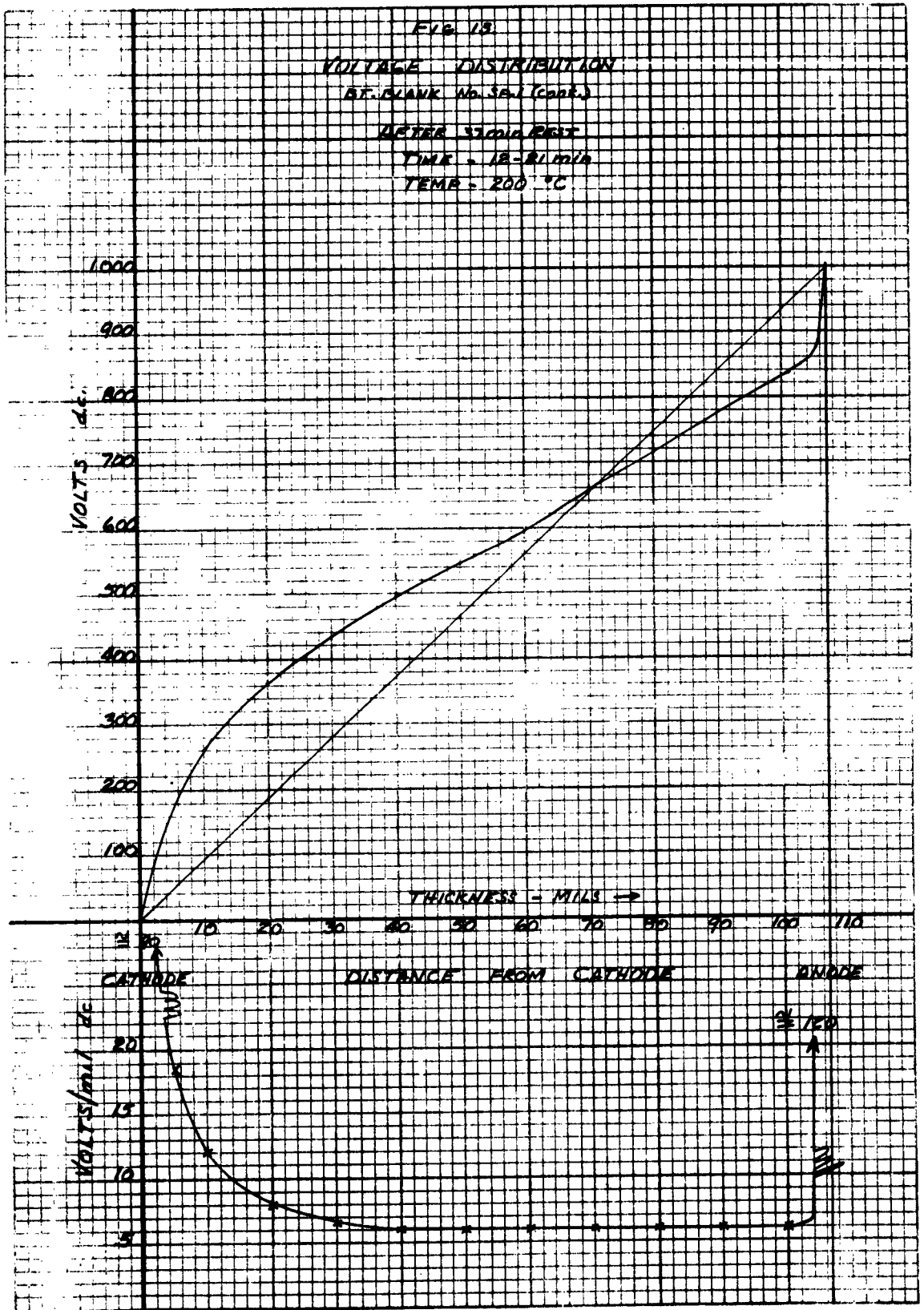


FIG. 14

VOLTAGE DISTRIBUTION
OF BLANK No. SP-1 (Cont.)

AFTER 37 min. TEST
TIME - 25-35 min
TEMP - 200 °C

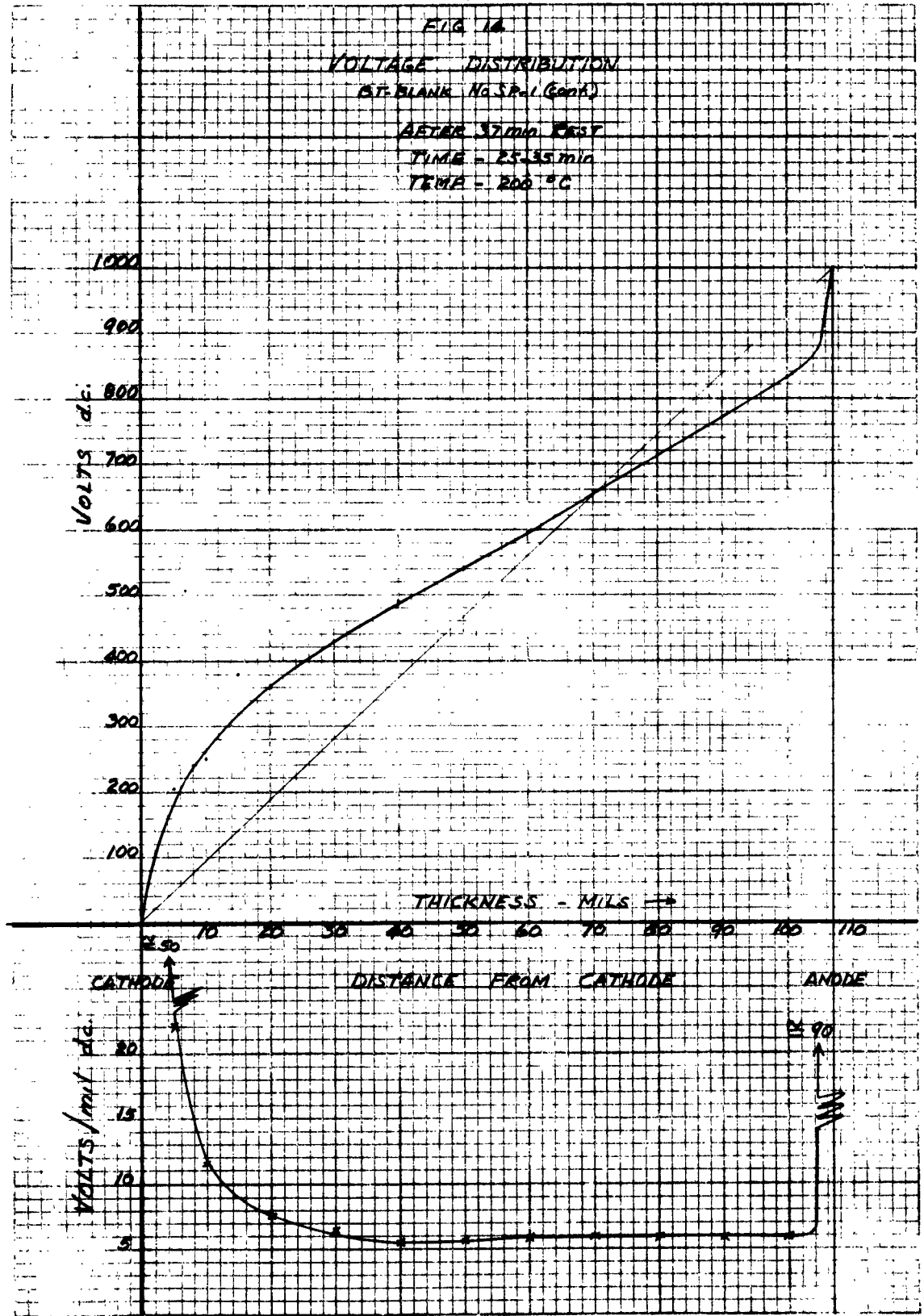


FIG 15
VOLTAGE DISTRIBUTION
AT-BLANK No. SP-1 (Cont.)

AFTER 37 min REST

TIME - 2.3-2.5 HRS

TEMP - 200 °C

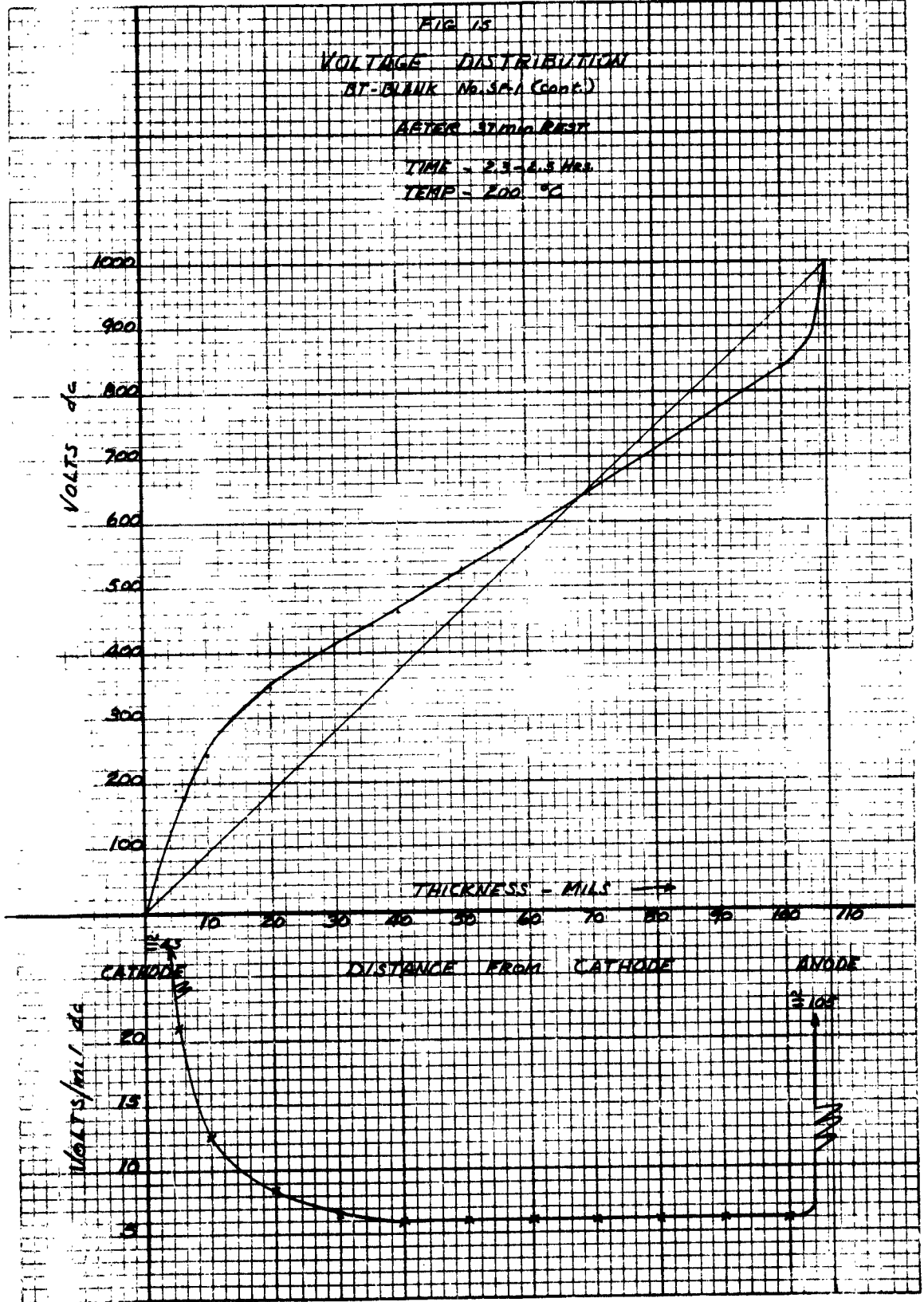


FIG 16

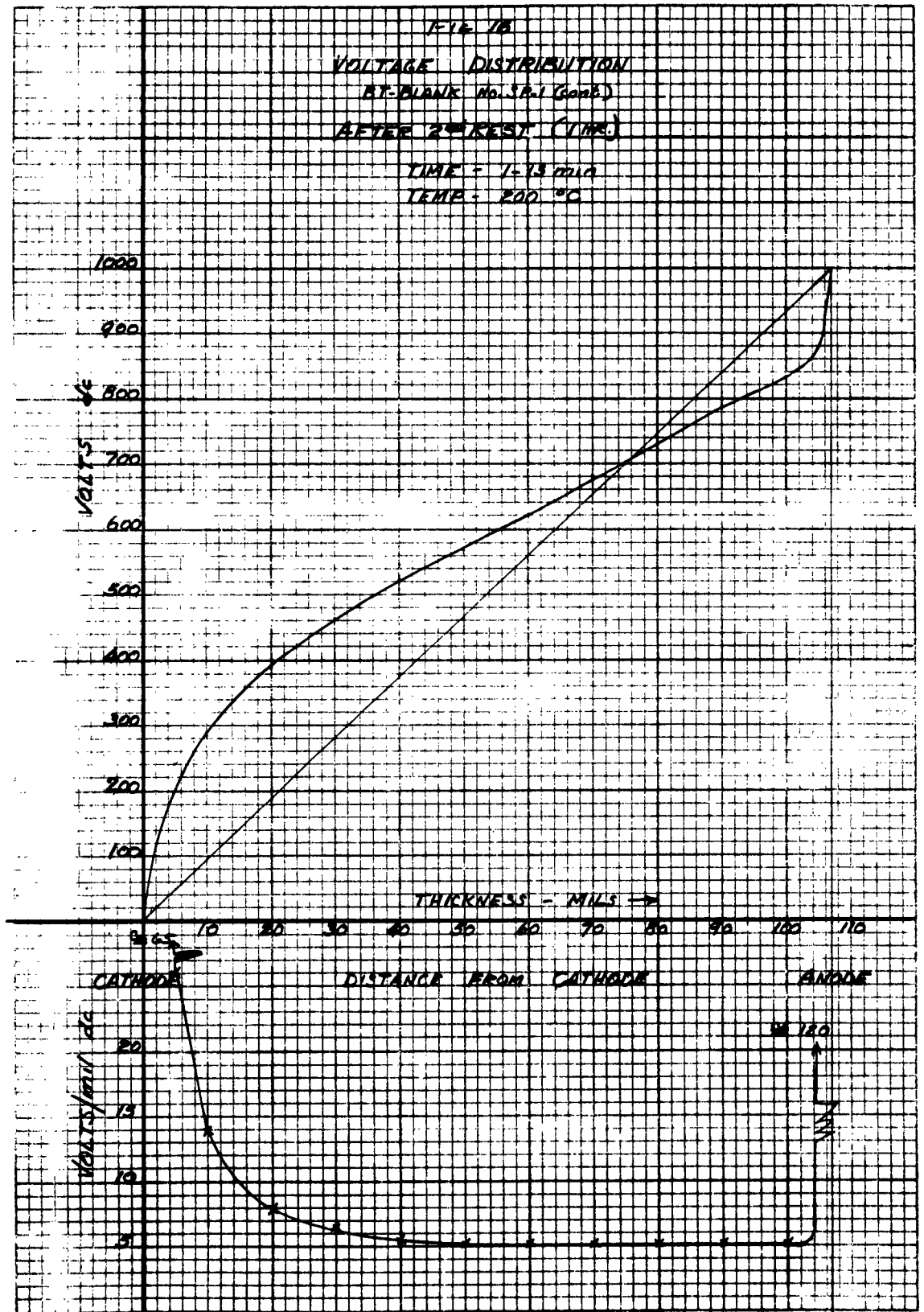
VOLTAGE DISTRIBUTION

BT-BLANK No. 5A-1 (cont.)

AFTER 2nd REST. (VHR.)

TIME - 1-13 min

TEMP - 200 °C



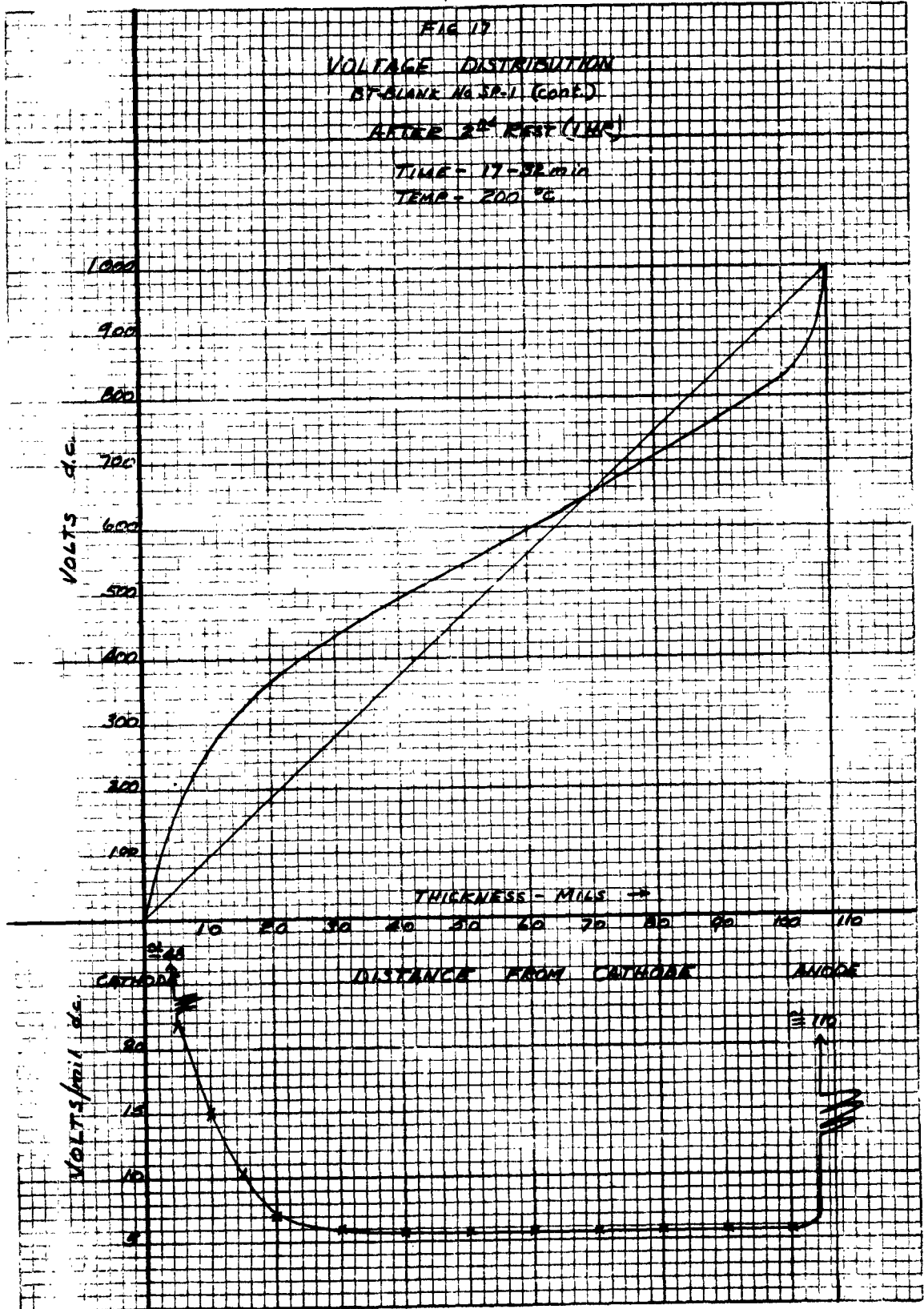


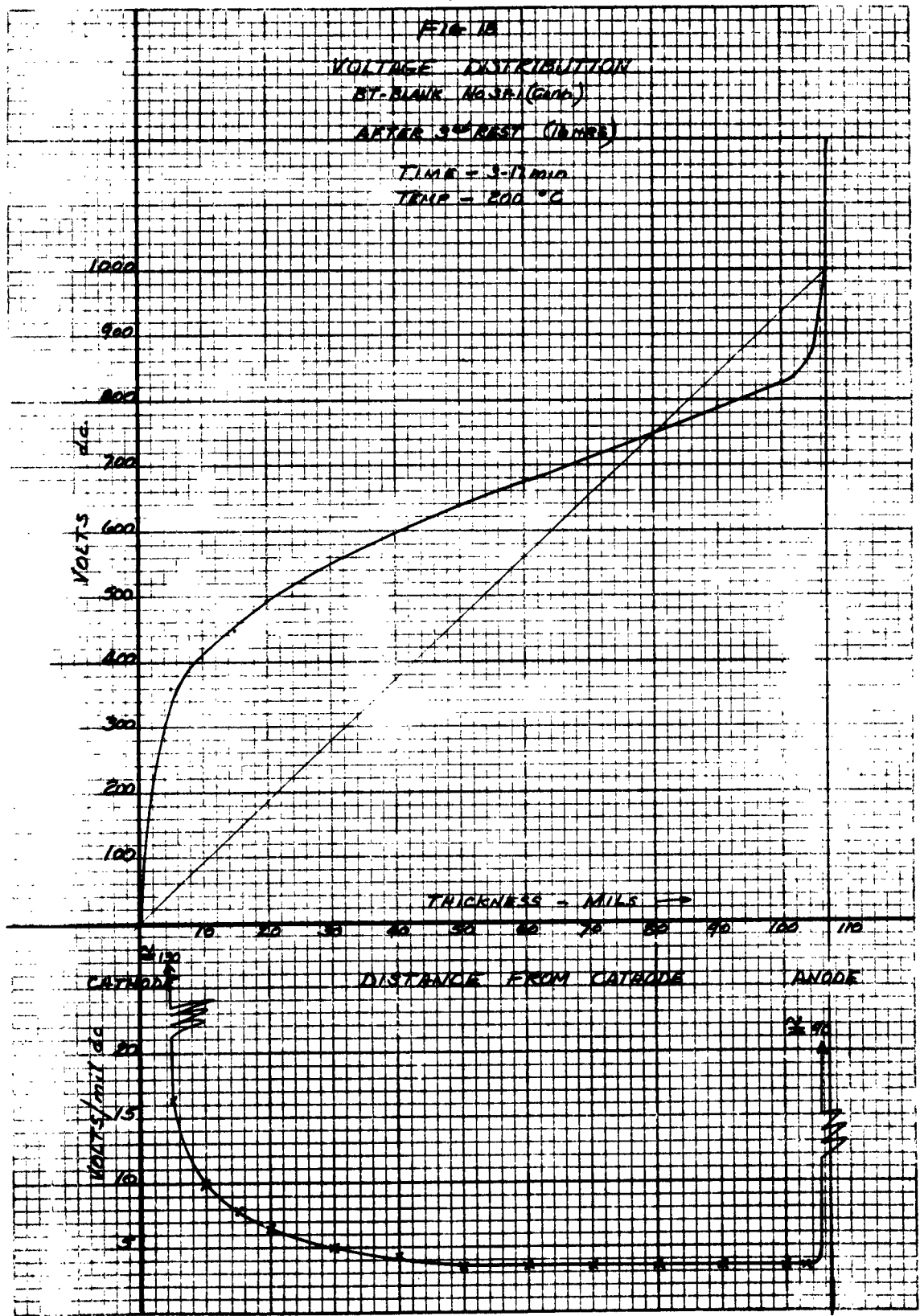
FIG 1B

VOLTAGE DISTRIBUTION
BT-BLANK NO. 381 (GAP)

AFTER 3rd REST (10 MRS)

TIME = 3-11 min

TEMP = 200 °C



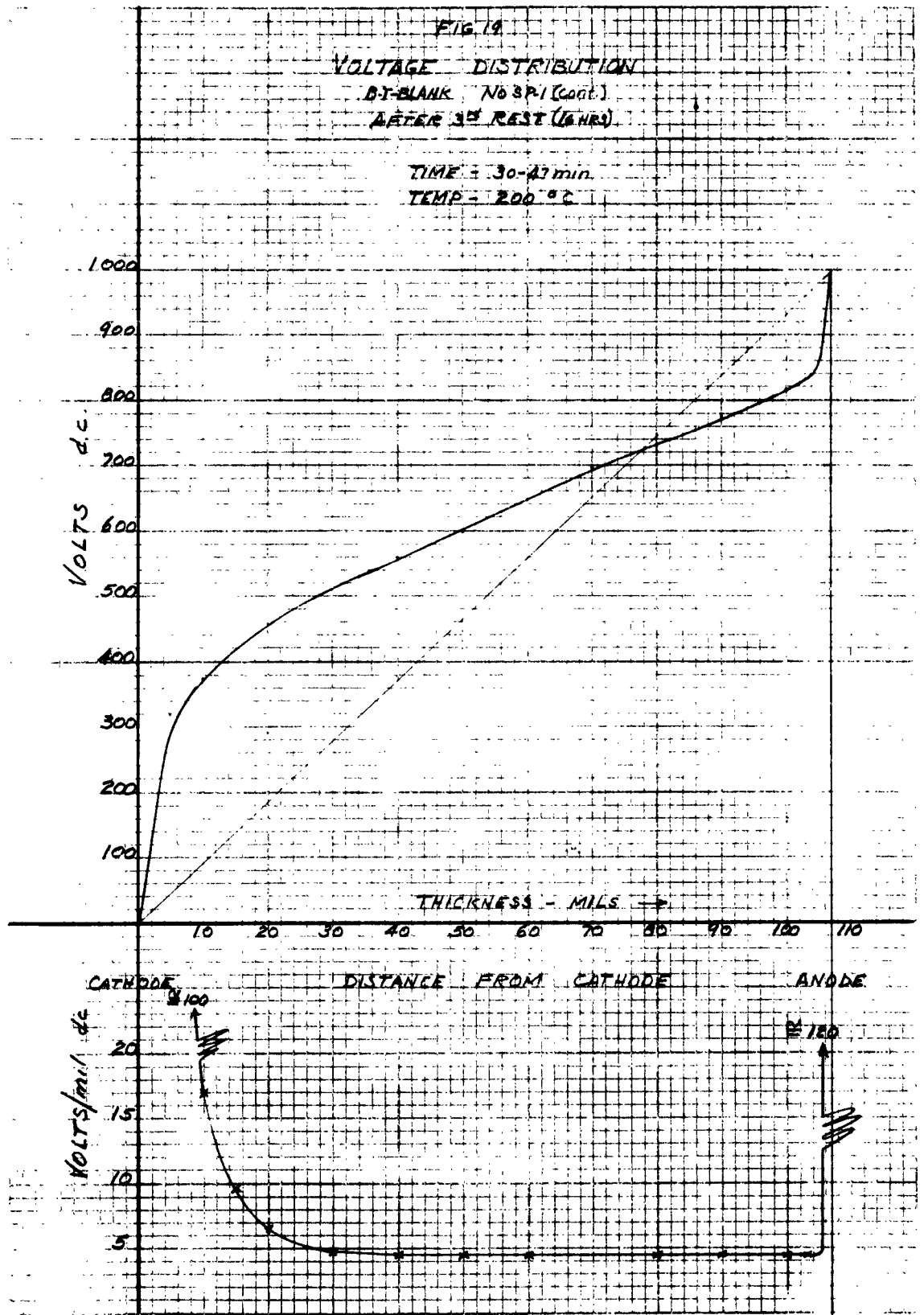
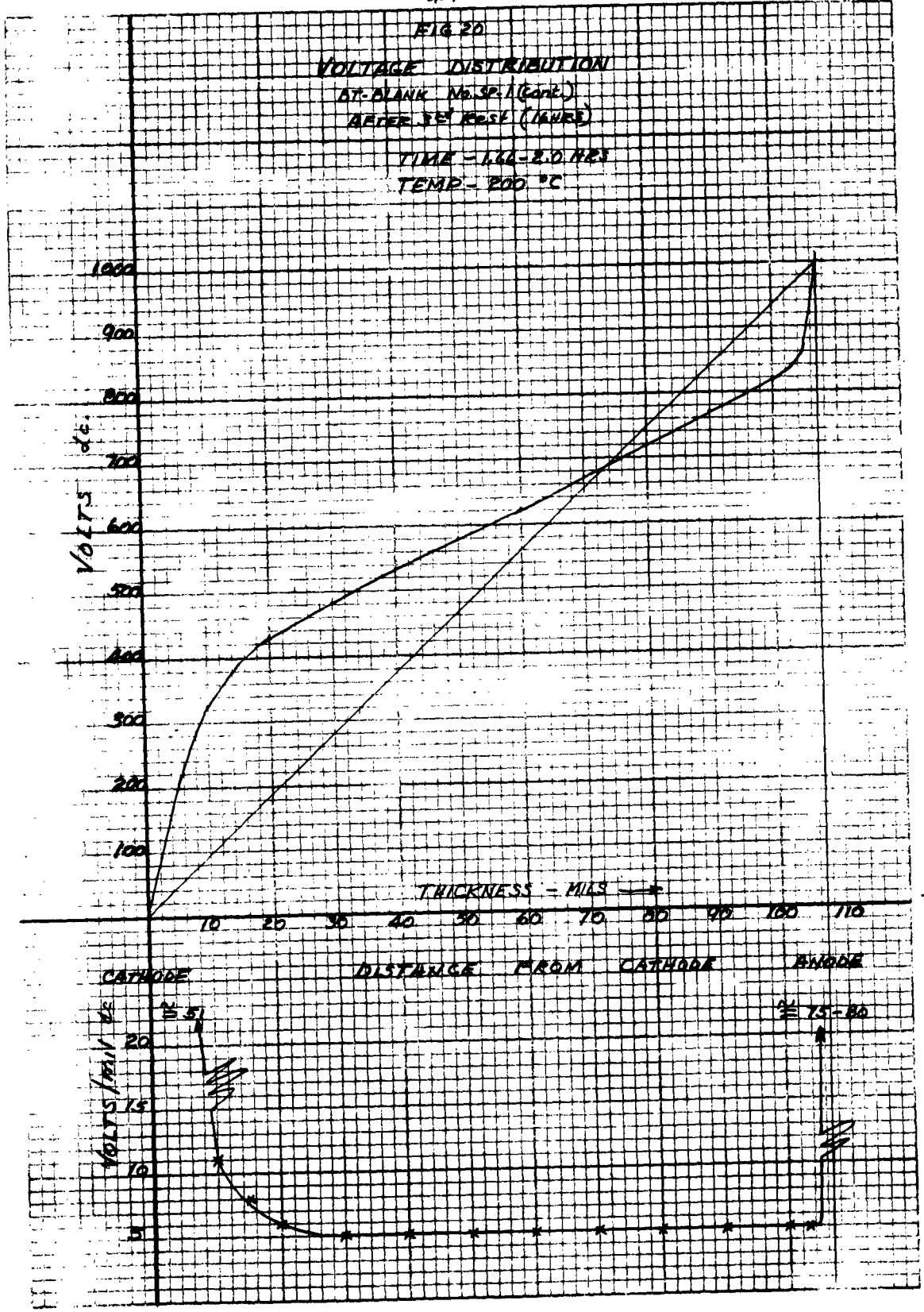


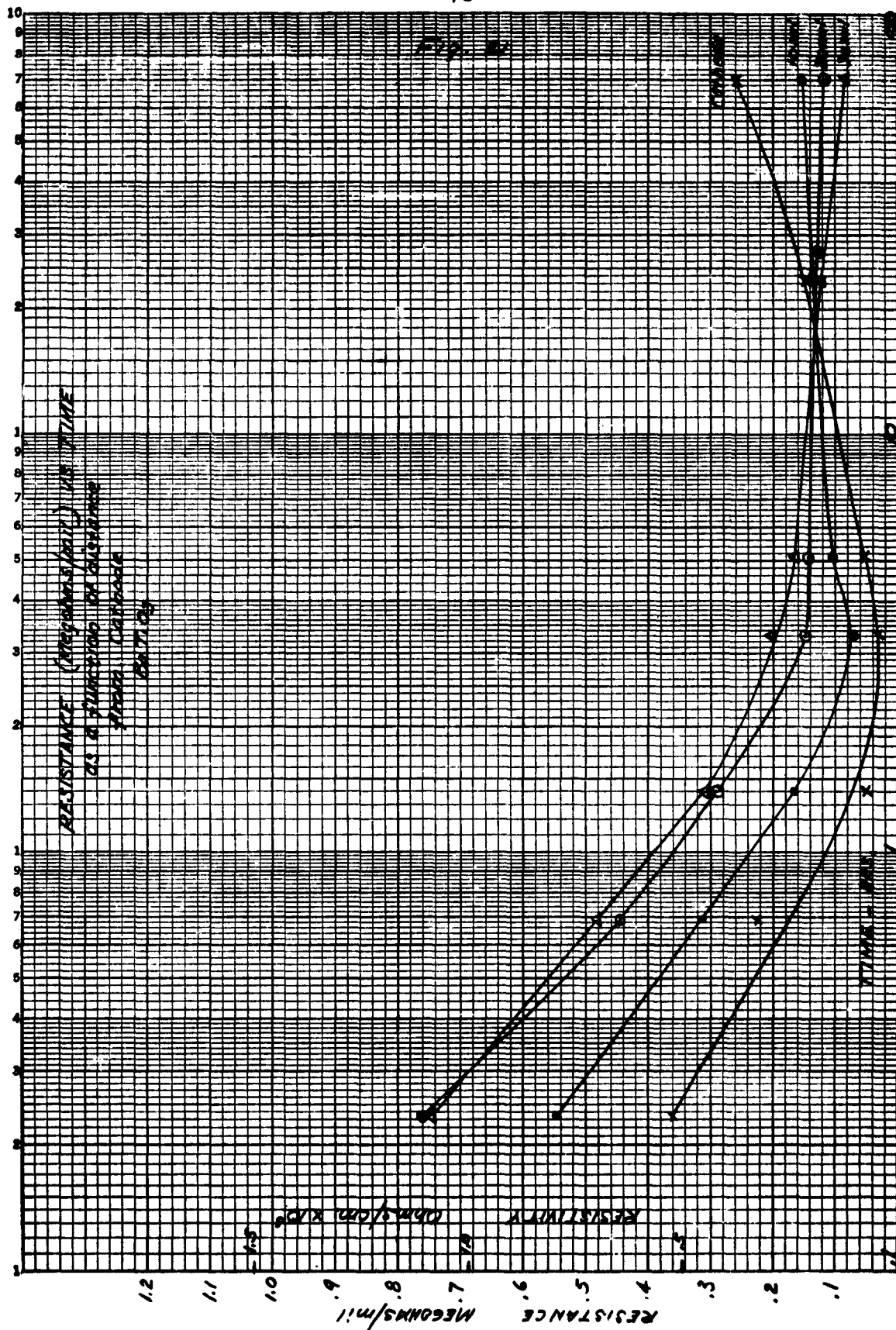
FIG 20

VOLTAGE DISTRIBUTION

DT-BLANK No. SP. 1 (Cont.)
AFTER 1st REST (16URS)

TIME - 1.66-2.0 HRS
TEMP - 200 °C





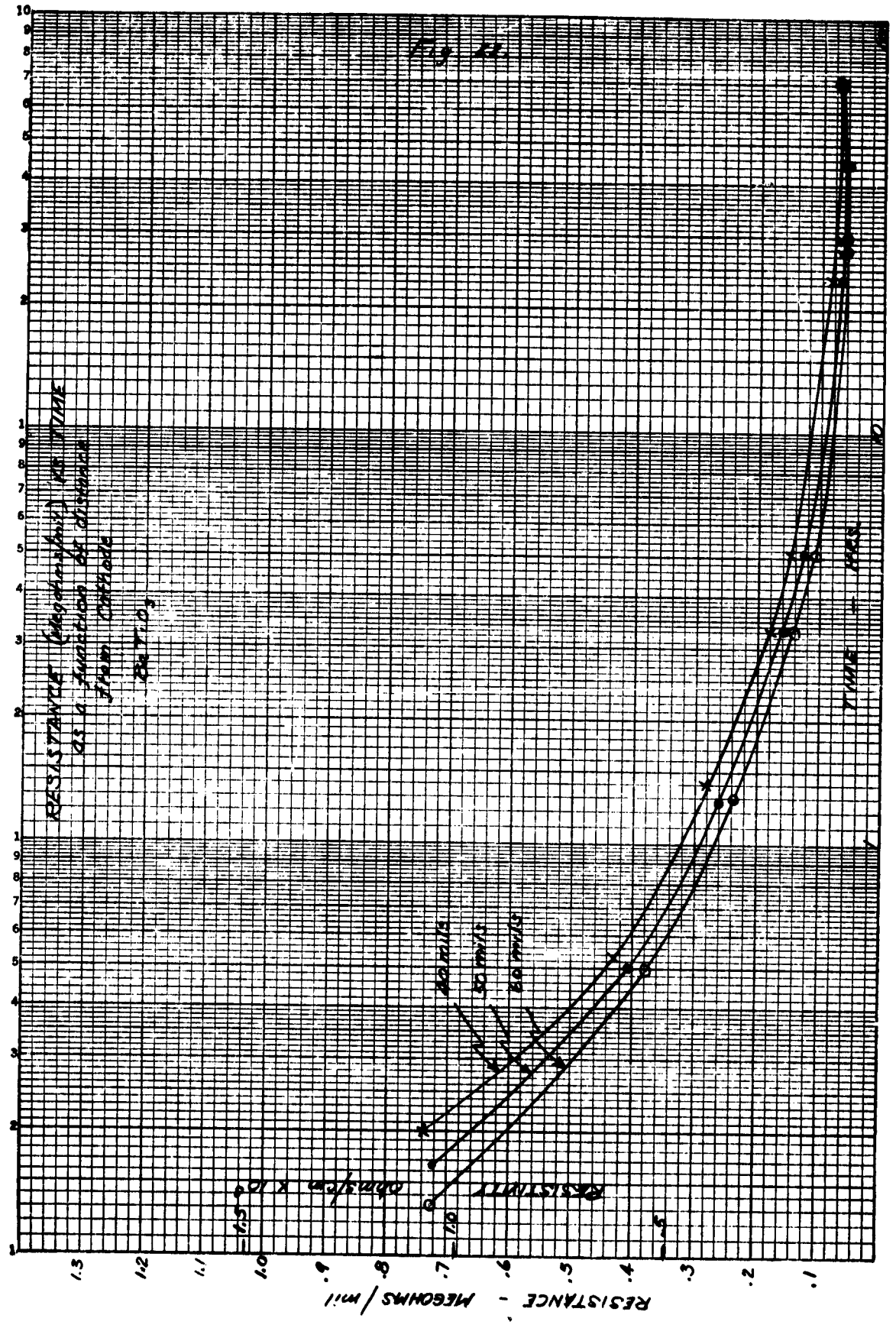
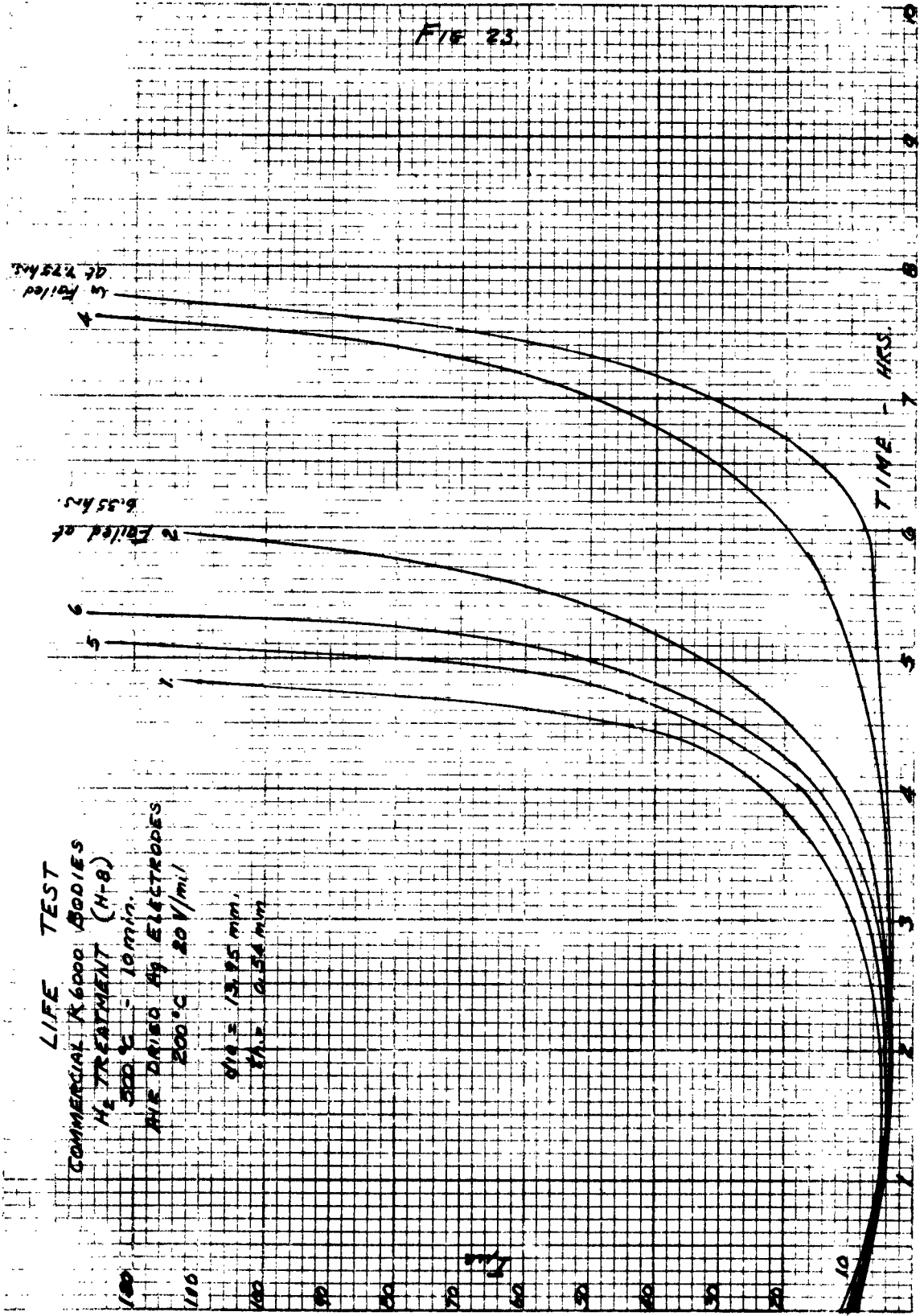


FIG 23

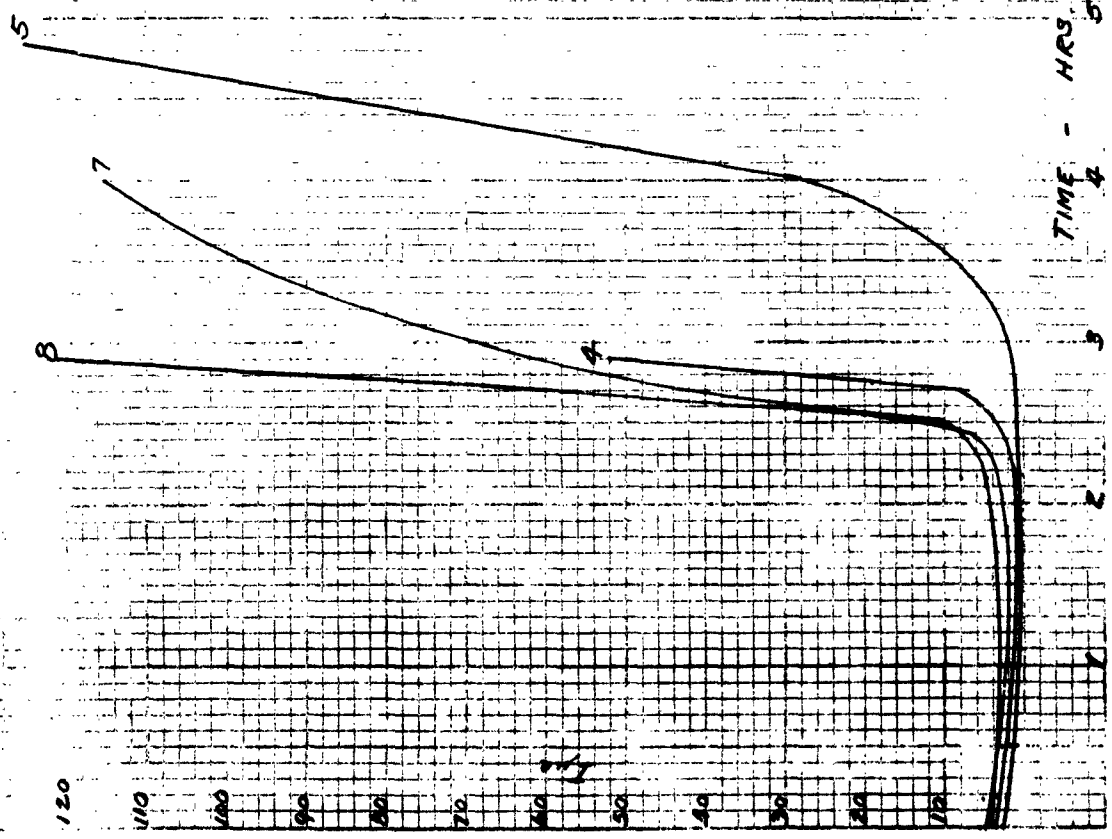


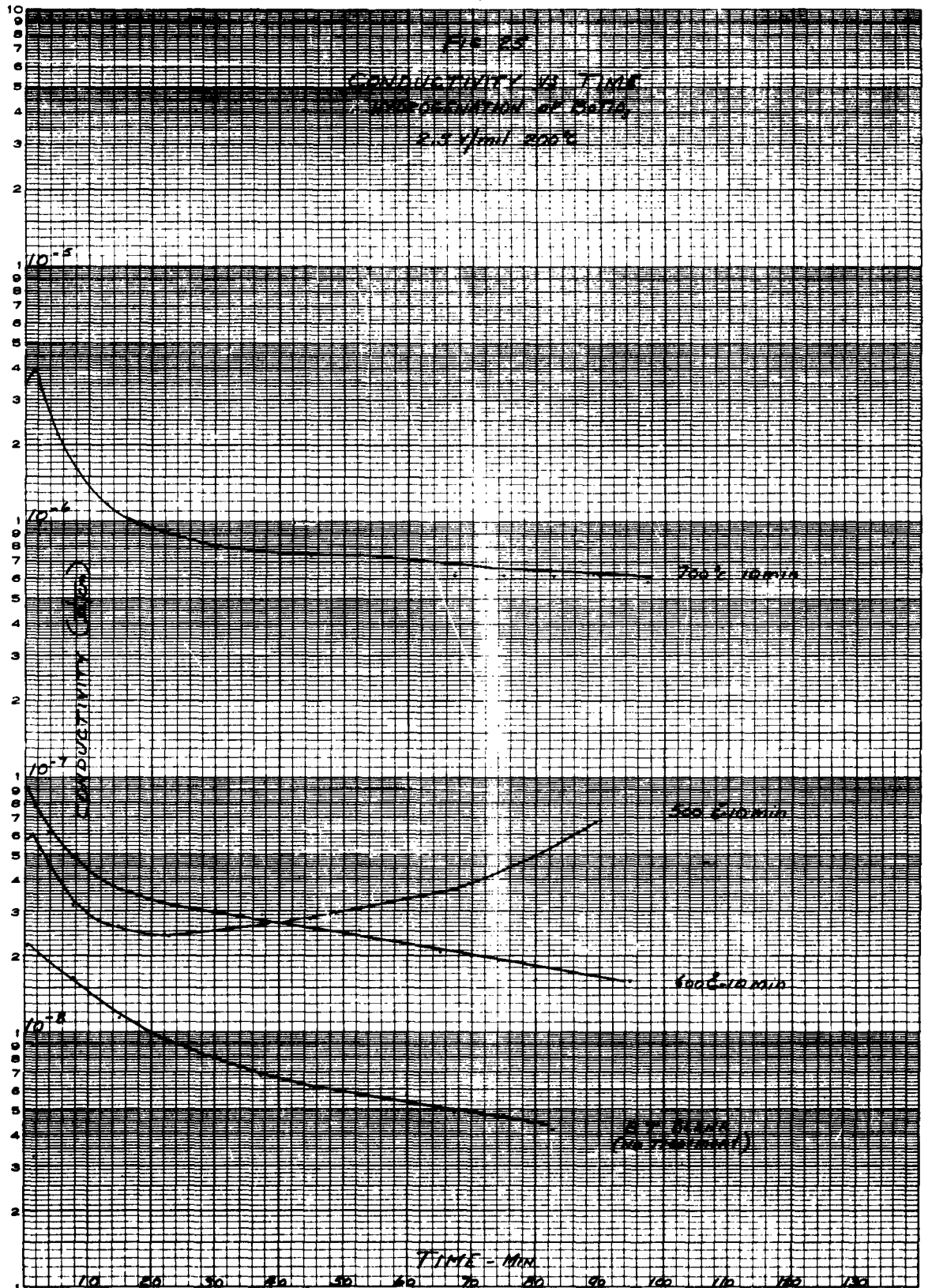
LIFE TEST
COMMERCIAL K 6000 BODIES
H₂ TREATMENT (H-8)
300% - 10 min.
AIR DRIED 49 ELECTRODES
200°C 20 V/mil
Ø = 1.25 mm
L = 0.54 mm

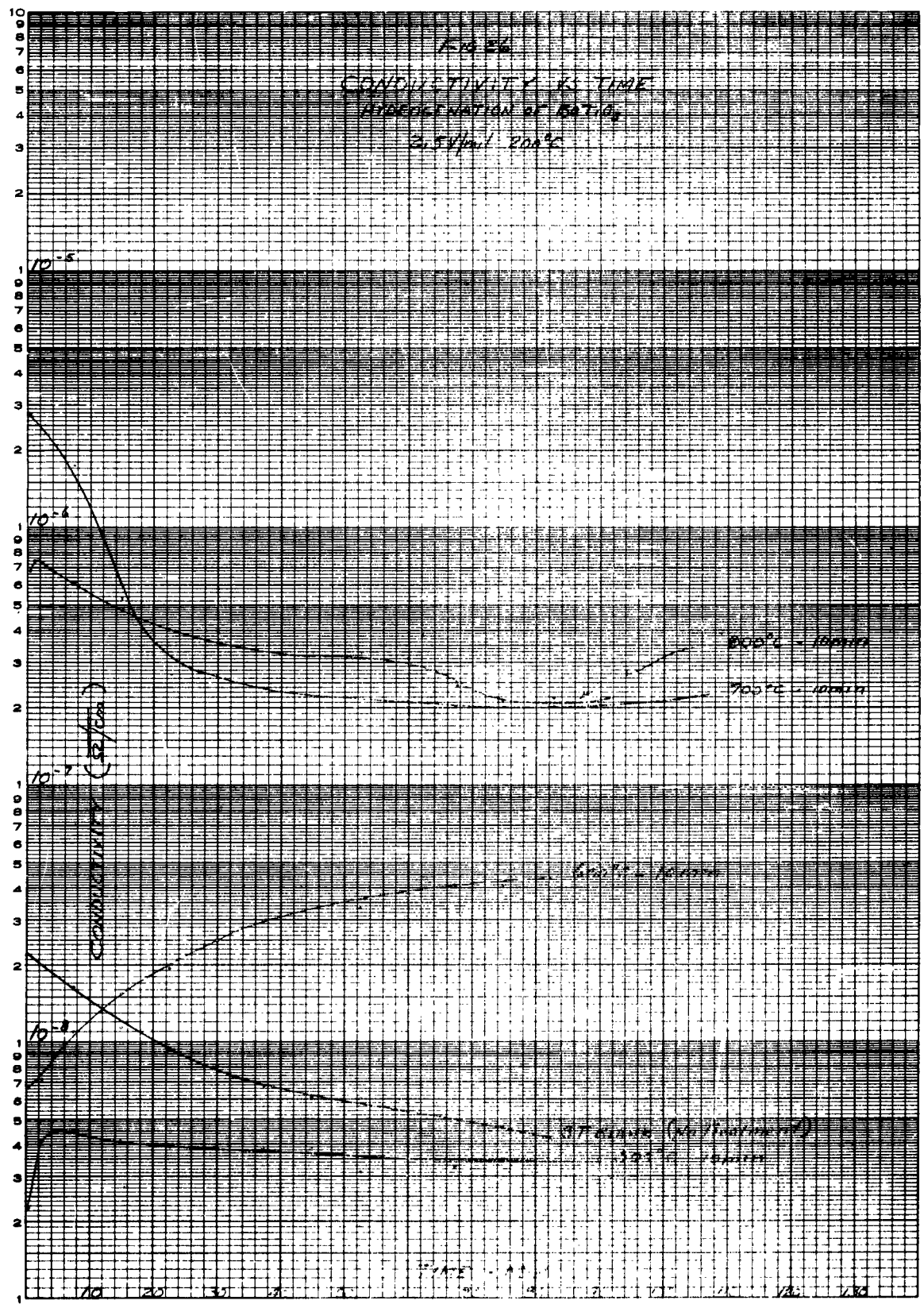
FIG. 2A

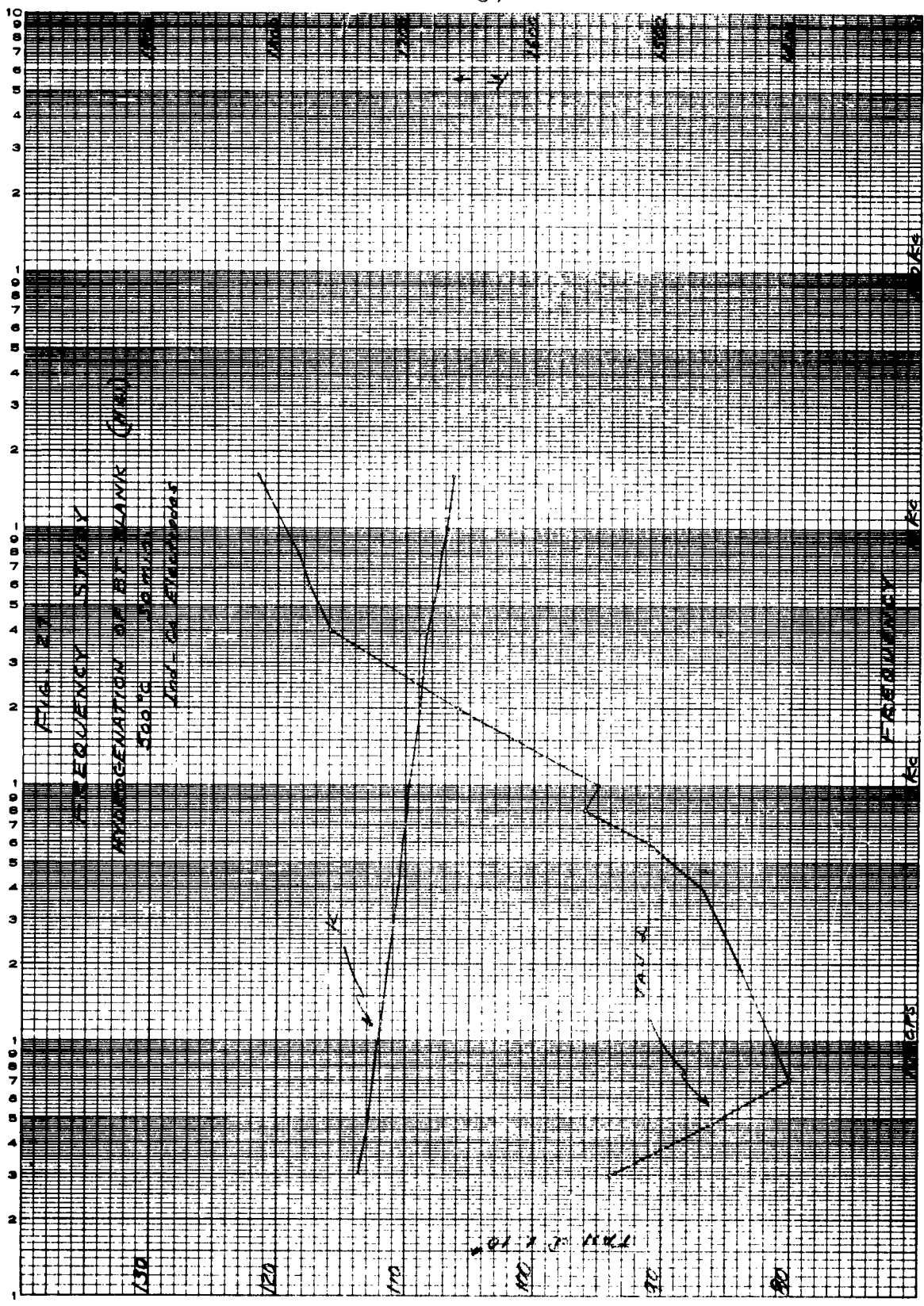
LIFE TEST
COMMERCIAL K6000 BODIES
NO TREATMENT

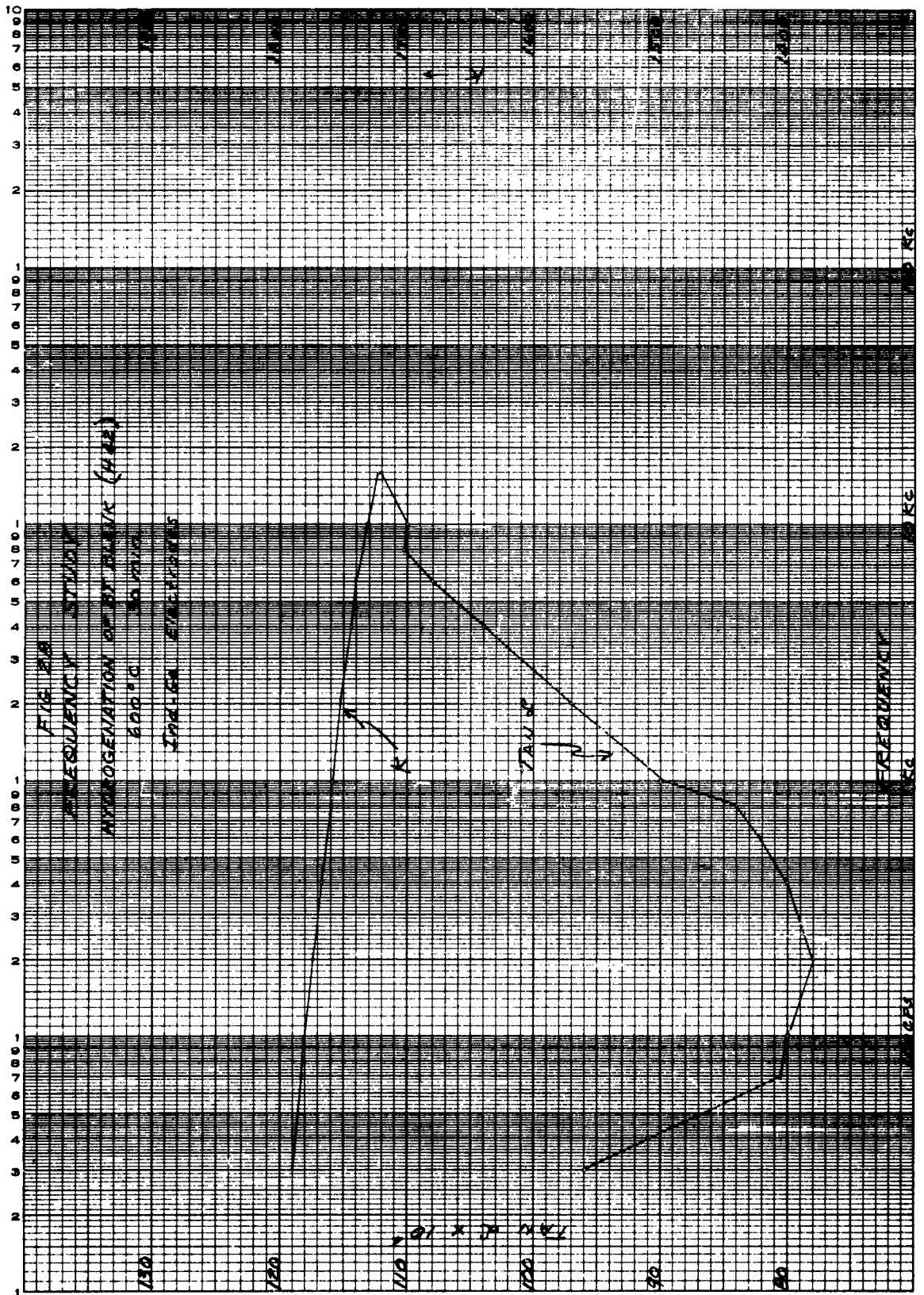
AIR DRIED Ag ELECTRODES
200°C 20 V/μm
dia. 13.95 mm
th. 0.54 mm

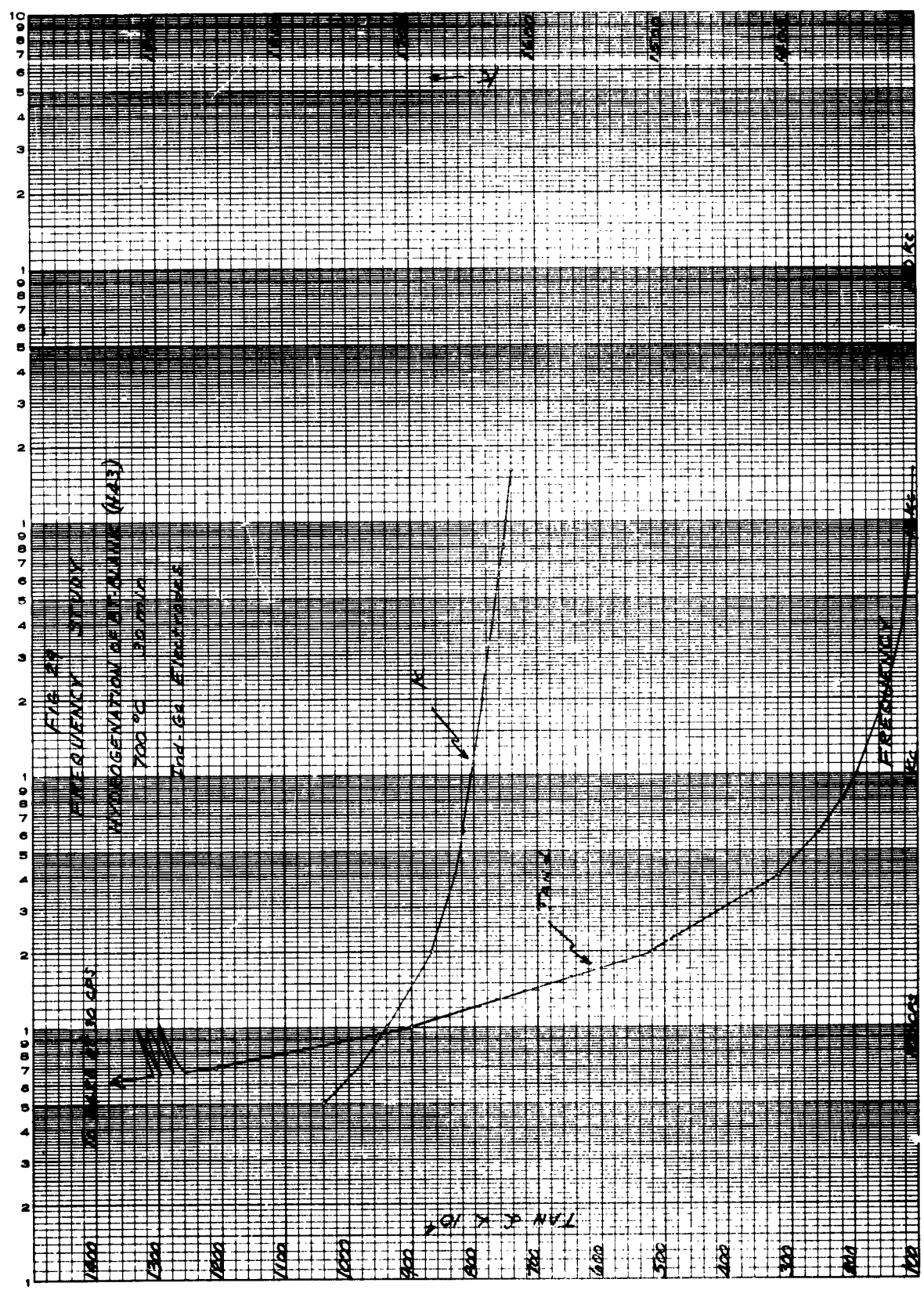


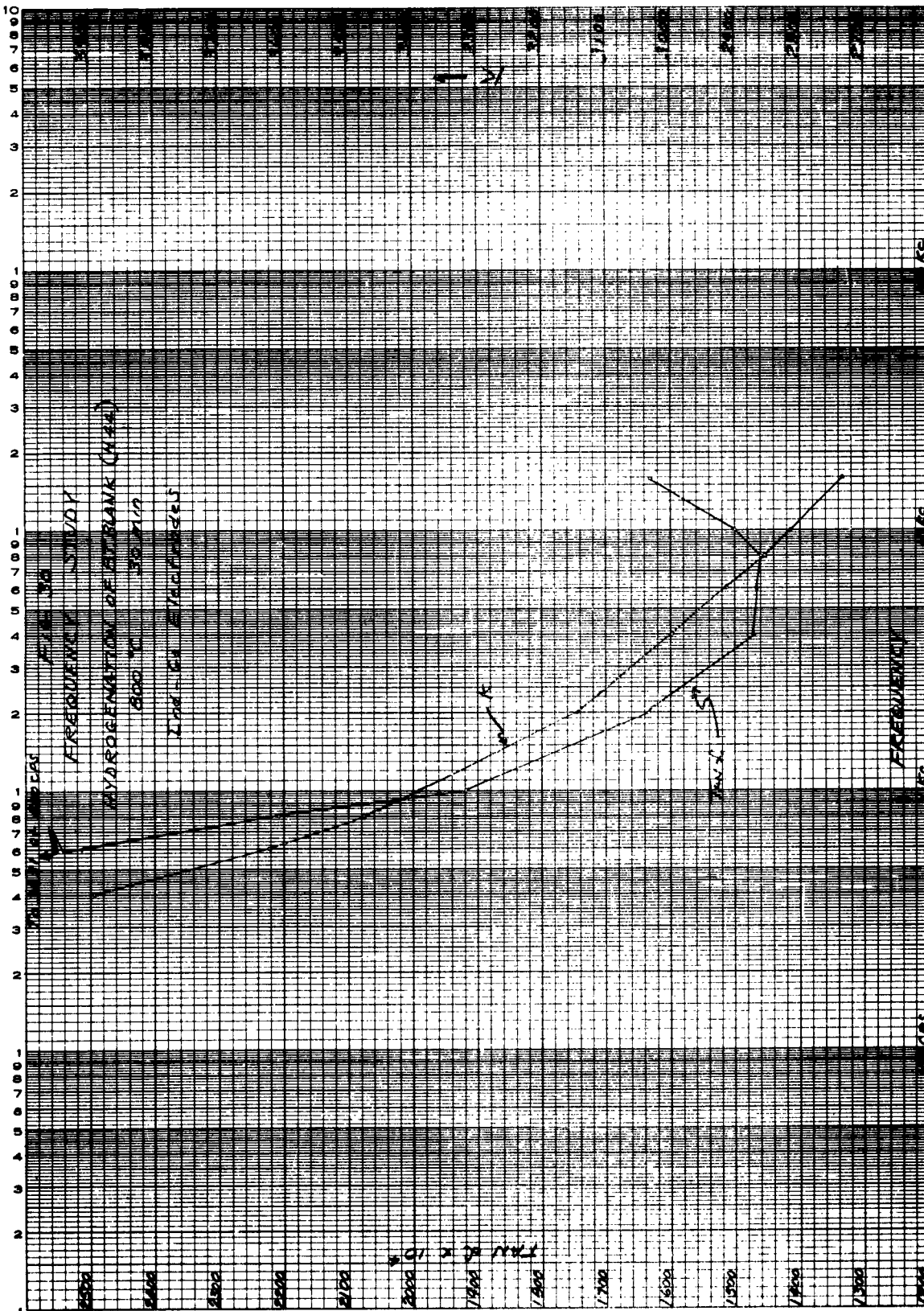


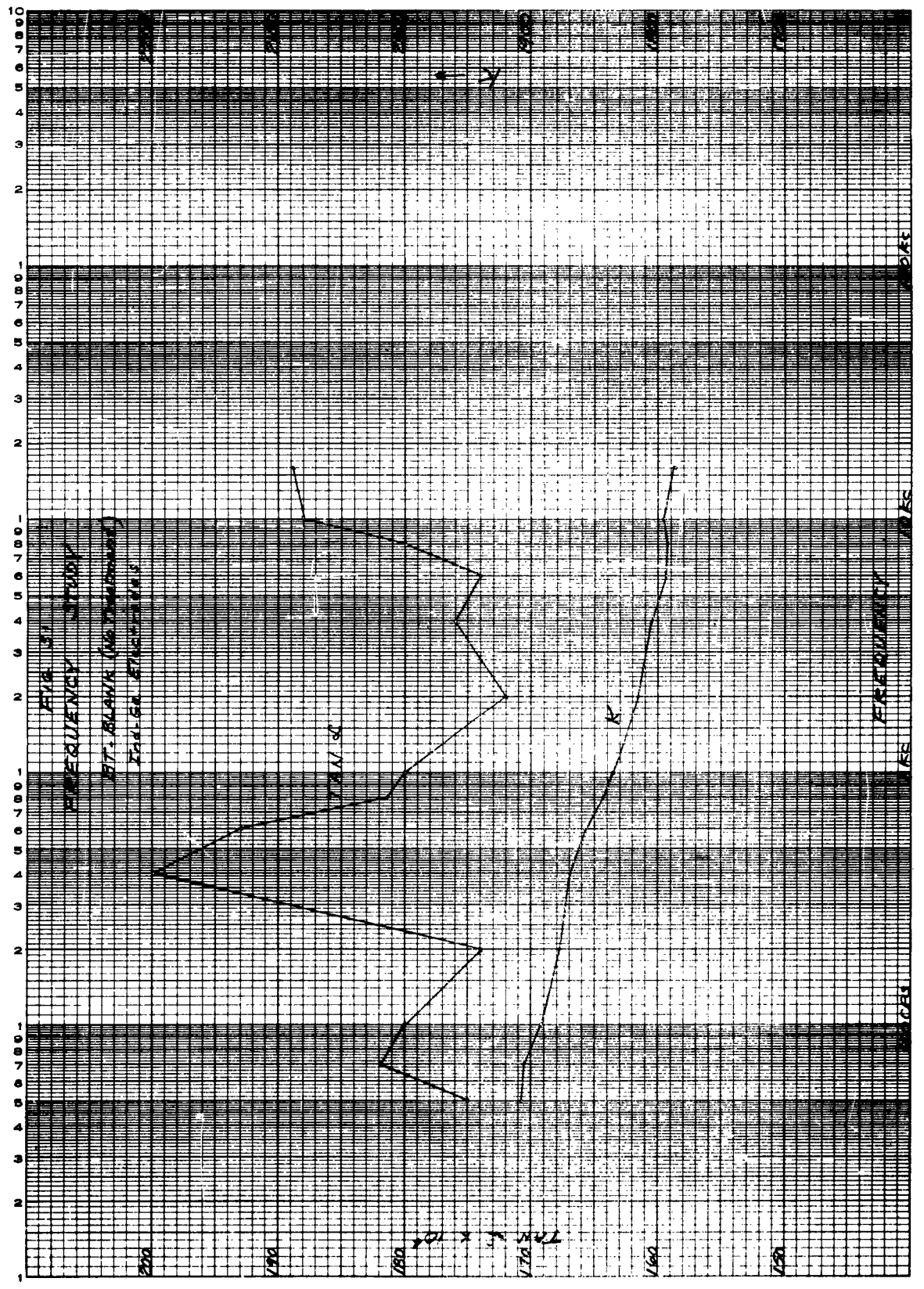








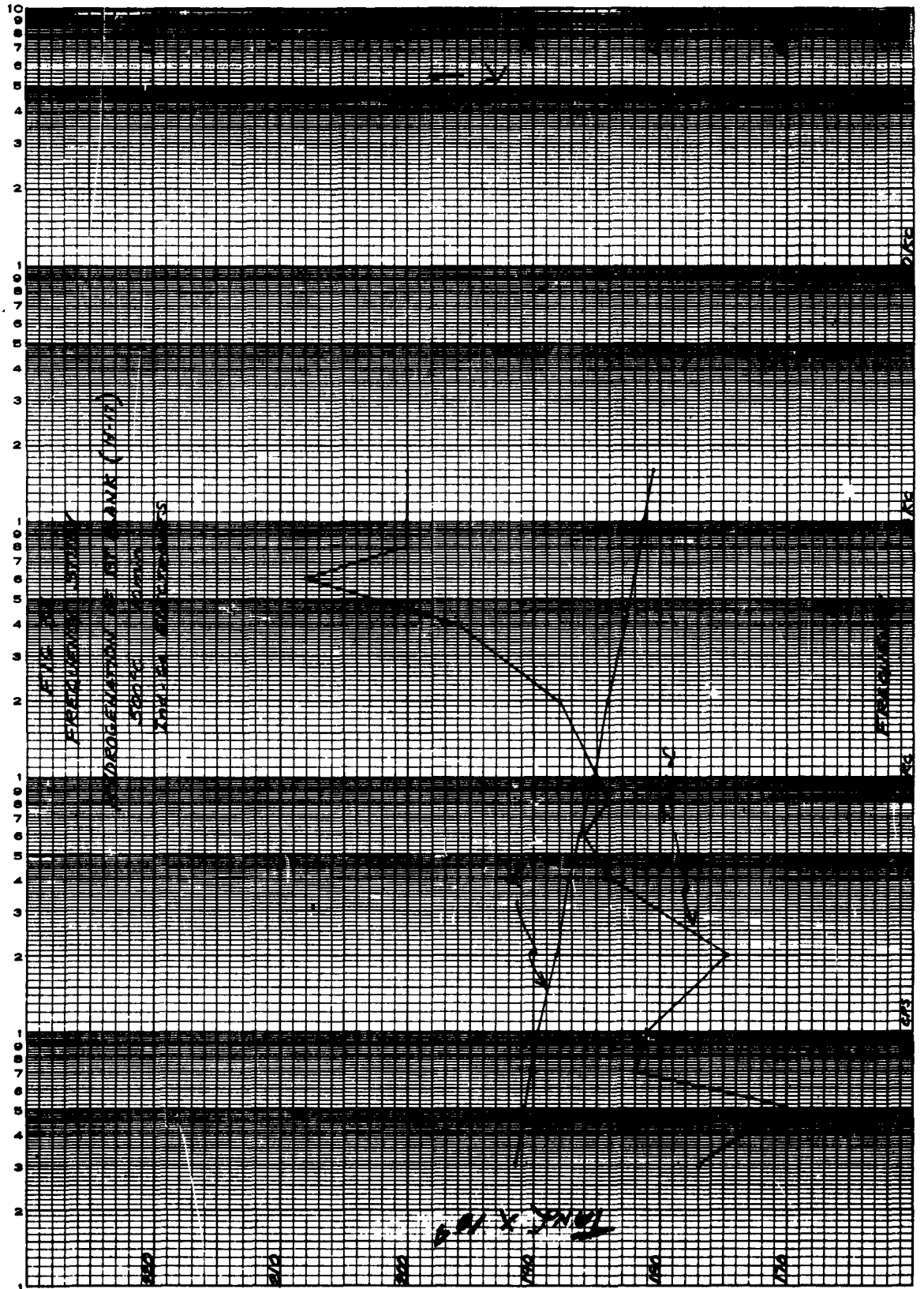


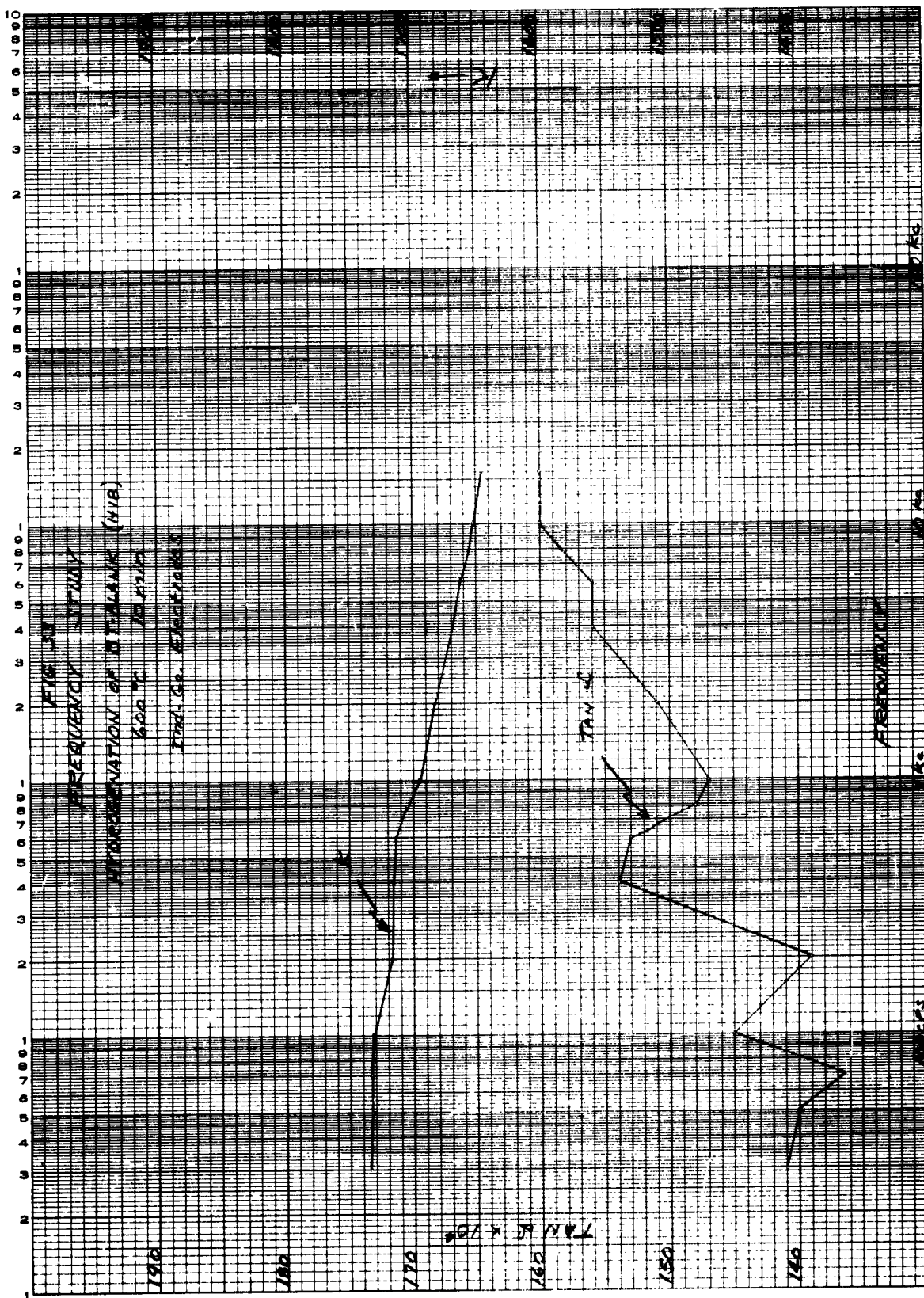


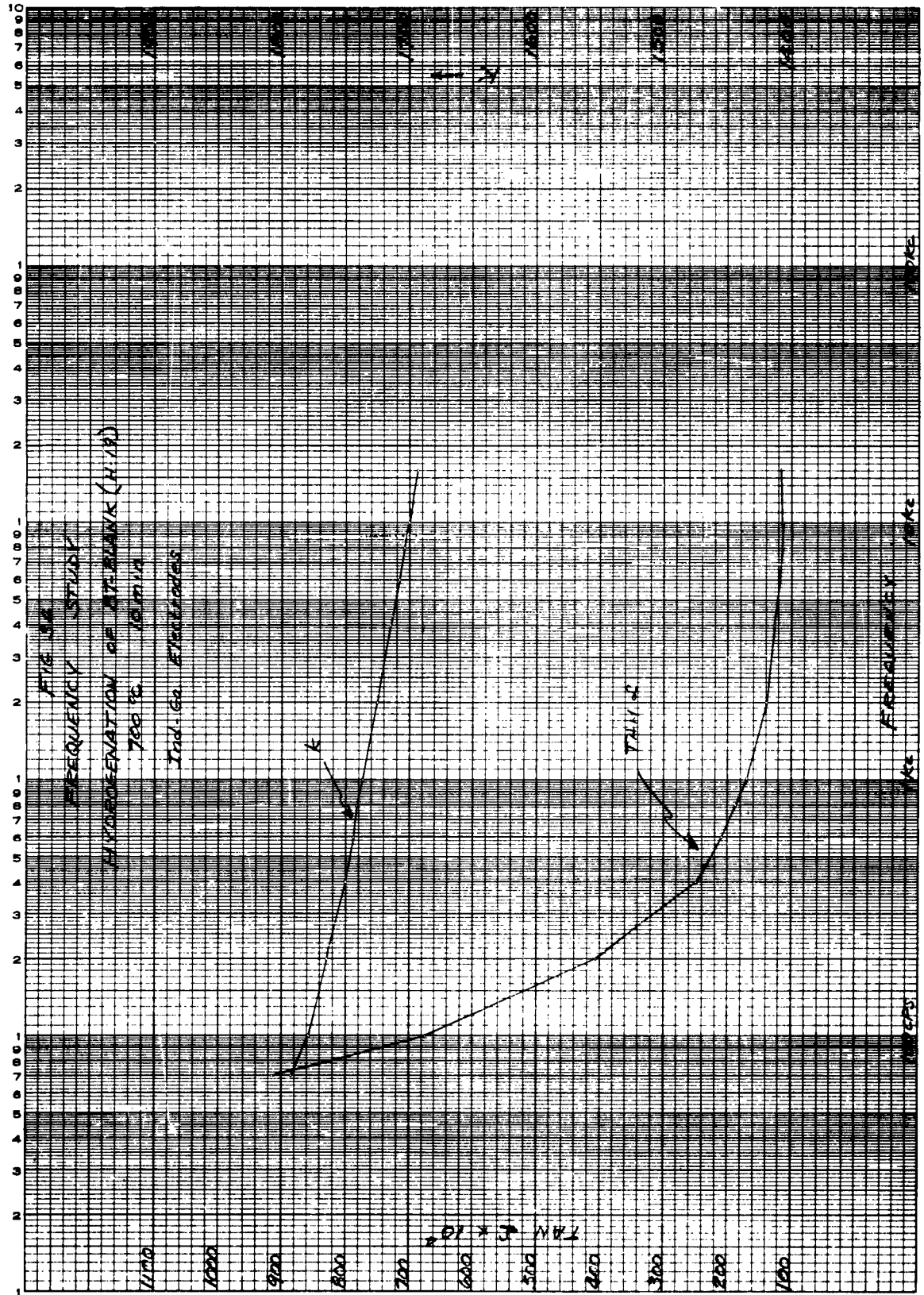
AYE ST
EMERGENCY STUDY
BT. BLANK (MATHS)

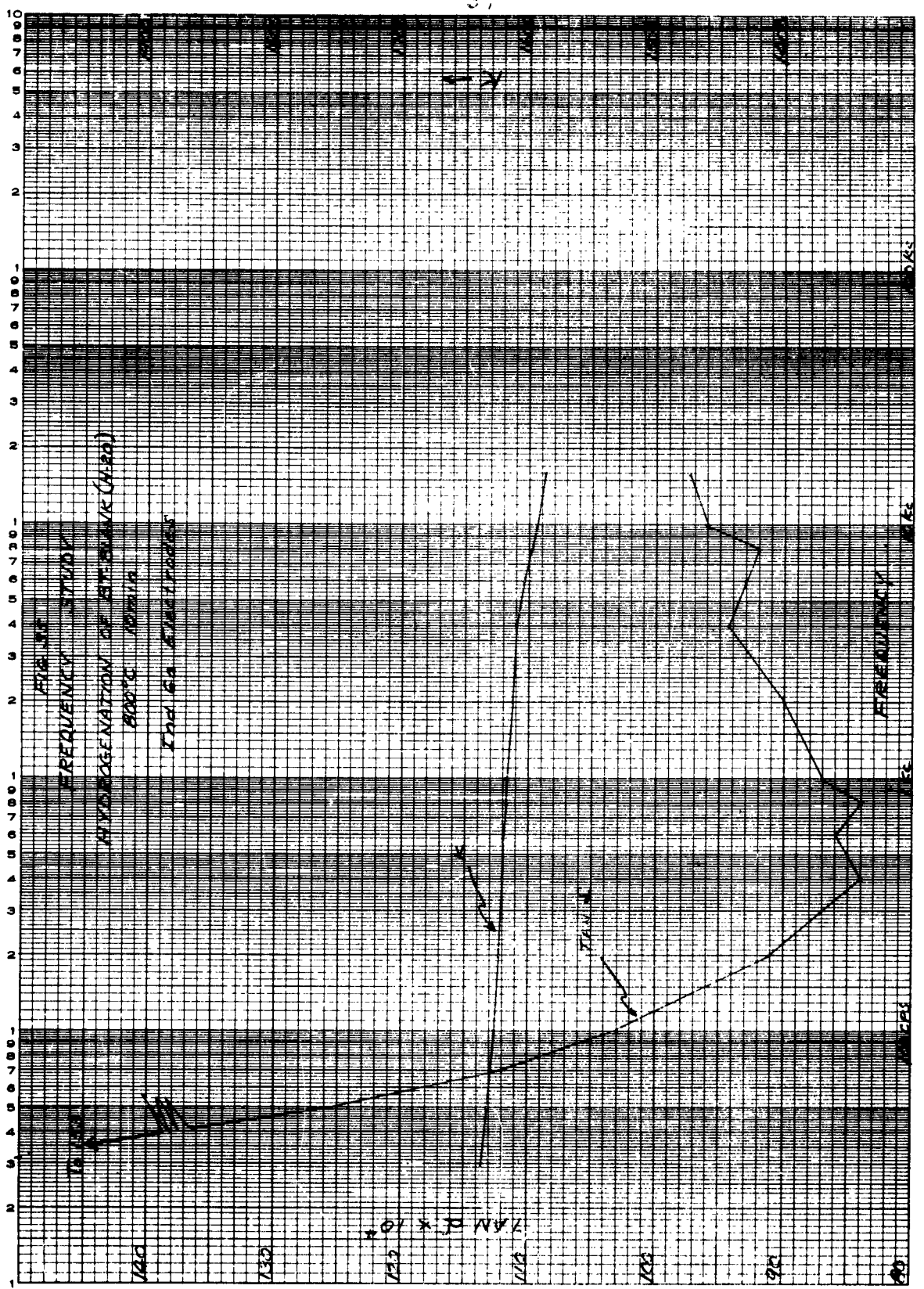
FREQUENCY

TAN δ x 10⁴







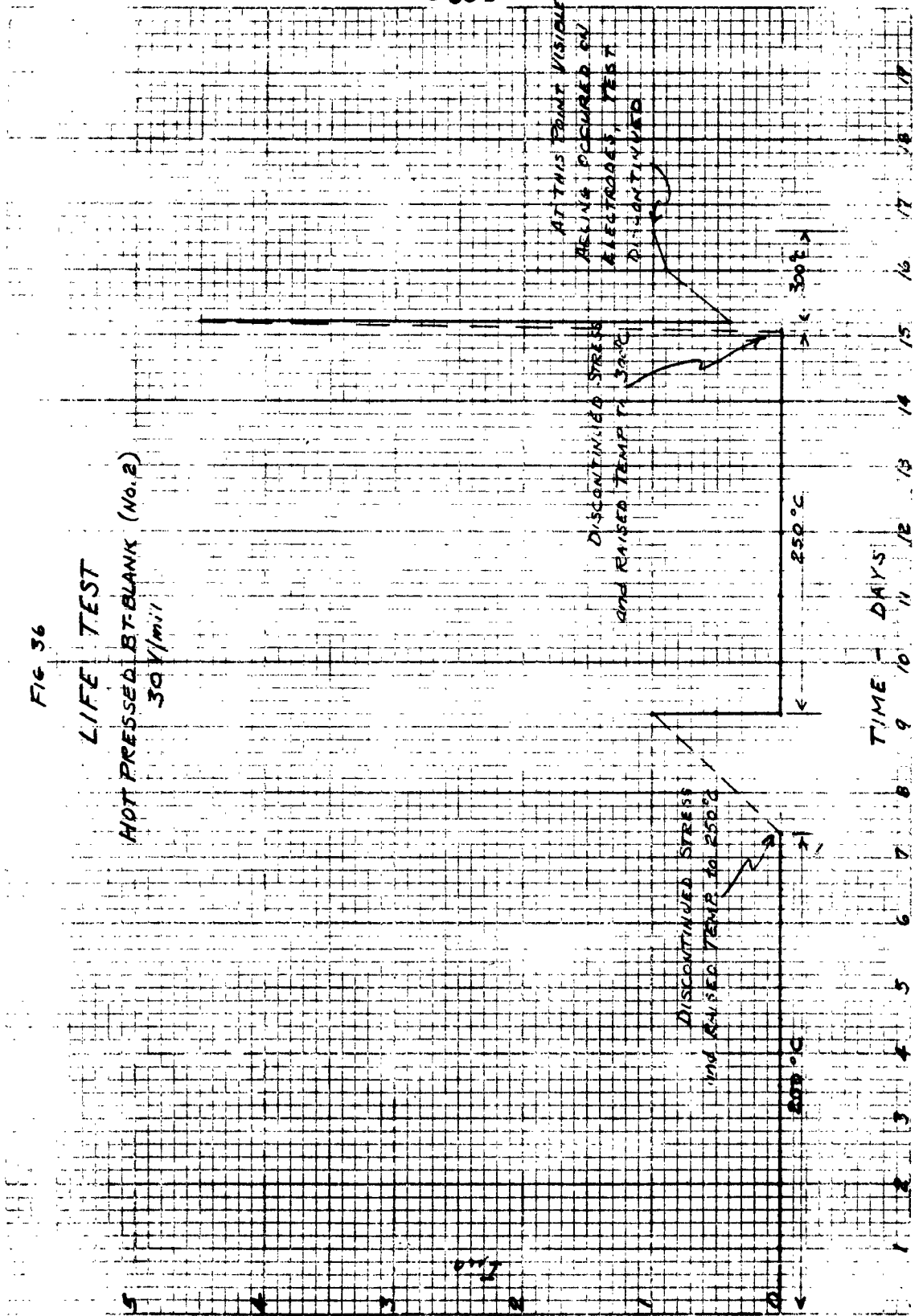


JAN 21 1954
 100 x 10^4

FIG 36

LIFE TEST

HOT PRESSED BT-BLANK (No. 2)
30 V/mil



V. IDENTIFICATION OF PERSONNEL

W. R. Buessem	Project Director	1/6 Time
W. A. Weyl	Project Advisor	1/6 Time
P. A. Marshall, Jr.	Project Scientist	5/6 Time
S. J. Beyer	Technical Asst.	Full Time
H. Roman	Technical Asst.	Full Time
W. Russell	Technical Asst.	Full Time

<p>AD Div. 14/2, 25/6.....</p> <p>Linden Laboratories, Inc. First Quarterly Report on CRYSTAL CHEMISTRY OF CERAMIC DIELECTRICS</p> <p>W. R. Buessem, P. A. Marshall, Jr. Report No. 17, 15 March 1963 65 pgs. incl. illus. tables</p> <p>Contract DA-36-039 AMC-00107(E), continuation of Contract DA-36-039 SC-78912</p> <ol style="list-style-type: none"> 1. Voltage Distribution in BaTiO₃ under DC Stress 2. Effect of H₂ and H₂O on Electrical Properties of BaTiO₃ 3. Hot Pressed Samples, Life Test 	<p>UNCLASSIFIED</p> <ol style="list-style-type: none"> 1. Ceramic Capacitor — Electrical Properties 2. Ceramic Materials—Degradation 3. Contract DA-36-039 AMC-00107(E) 	<p>AD Div. 14/2, 25/6.....</p> <p>Linden Laboratories, Inc. First Quarterly Report on CRYSTAL CHEMISTRY OF CERAMIC DIELECTRICS</p> <p>W. R. Buessem, P. A. Marshall, Jr. Report No. 17, 15 March 1963 65 pgs. incl. illus. tables</p> <p>Contract DA-36-039 AMC-00107(E), continuation of Contract DA-36-039 SC-78912</p> <ol style="list-style-type: none"> 1. Voltage Distribution in BaTiO₃ under DC Stress 2. Effect of H₂ and H₂O on Electrical Properties of BaTiO₃ 3. Hot Pressed Samples, Life Test 	<p>UNCLASSIFIED</p> <ol style="list-style-type: none"> 1. Ceramic Capacitor — Electrical Properties 2. Ceramic Materials—Degradation 3. Contract DA-36-039 AMC-00107(E)
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