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RESEARCH AND DEVELOPMENT PROGRAM
INTRINSIC RELIABILITY
SUBMINIATURE CERAMIC CAPACITORS

EIGHTH QUARTERLY PROGRESS REPORT

PERIOD: 1 MARCH 1964 - 31 MAY 1964

TO

U. S. ARMY SIGNAL RESEARCH & DEVELOPMENT LABORATORY

FORT MONMOUTH, NEW JERSEY

CONTRACT NO. DA-36-039-SC-90705

D. A. PROJECT NO. 3A99-15-001

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RESEARCH AND DEVELOPMENT PROGRAM

INTRINSIC RELIABILITY

SUBMINIATURE CERAMIC CAPACITORS

Eighth Quarterly Report

Period: 1 March 1964 - 31 May 1964

Object of Study: To conduct investigations leading to the approaches for the attainment of high reliability in subminiature ceramic capacitors and the determination of failure rate as a function of voltage and temperature.

Contract No. DA-36-039-SC-90705
D. A. Project No. 3A99-15-001

Controlling Specifications:

Signal Corps Technical Guidelines, "Reliability Long Life Component Studies," 3 November 1961

Signal Corps Technical Requirements No. SCL-2101N, 14 July 1961

Report Prepared by:

J. H. D. Folster
T. I. Prokopowicz
W. A. Tatem

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SECTION 1

PURPOSE

The purpose of this contract is to carry out research work for a period of 30 months commencing June 1, 1962, and ending November 30, 1964, involving investigations leading to approaches to the attainment of high reliability in subminiature ceramic capacitors and the determination of failure rate as a function of voltage and temperature.

In particular, this involves the following:

(1) Construction of a model or theory to predict failure mechanisms and failure rates as a function of voltage and temperature.

(2) Development and evaluation of a short-term test to eliminate early failures effectively without shortening the time to the wearout mode of failure.

(3) A determination of the failure rate as a function of voltage and temperature through large-scale life testing. From the data thus obtained, derating curves will be derived and overall failure rates for operating conditions will be estimated. The theory developed will be critically examined and refinements made.

(4) Compilation of quarterly progress reports in accordance with Signal Corps Technical Requirements No. SCL-2101N, dated 14 July 1961.

(5) Compilation of a final report in accordance with Signal Corps Technical Requirements No. SCL-2101N, dated 14 July 1961.

SECTION 2

ABSTRACT

A slight change in the ceramic composition of the C67 Case Size I MONOLYTHIC® capacitor has produced improved current stability which promises longer life. Characteristics of the improved capacitor with respect to leakage and voltage, temperature and time, are presented. An experiment has been started to examine the efficacy of a selection technique for the detection of potential early failures among the improved capacitors.

Life testing of 753 capacitors of the original version was continued. Pre-life test characteristics appear to support the life time formula developed during the seventh quarter, although not all the characteristics contained in the formula were determined on the individual capacitors before the start of testing because the formula was developed after the start of life testing. The importance of the elements contained in the life time formula is indicated by the results of other testing presented.

SECTION 3

PUBLICATIONS, LECTURES, REPORTS, AND CONFERENCES

(1) The Seventh Quarterly Report, covering the period December 1, 1963 - February 29, 1964, was submitted for U. S. Army Electronics Research and Development Agency approval. Approval was received, and the report was distributed per USAERDA instructions.

(2) On March 17, 1964, a conference was held at Fort Monmouth, New Jersey, to discuss progress to date and future work on this contract. Attending from USAERDA were Miss Jeanne Allen and Mr. H. L. Stout. Attending from Sprague Electric Company were Mr. T. I. Prokopowicz, Dr. D. M. Smyth, and Dr. G. A. Shirn.

(3) On April 29, 1964, a similar conference was held at Fort Monmouth. Attending from USAERDA were Miss Jeanne Allen and Mr. H. L. Stout. Attending from Sprague Electric Company were Mr. T. I. Prokopowicz and Mr. F. B. Schoenfeld.

SECTION 4

FACTUAL DATA

4.1 Construction of C67 Case Size I Monolythic Capacitors

The C67 Case Size I Monolythic capacitor has a construction of stacked ceramic dielectric layers 0.0025 in. thick and connected electrically in parallel. These layers are intimately bonded to each other through high-temperature sintering. The ceramic material is a barium titanate, which has a dielectric constant of about 2000 at room temperature, and which is stable to +10%, -15% between -55°C and 125°C. The capacitor is enclosed in a tubular case which is 0.25 in. long and 0.095 in. in diameter.

4.2 Characterization of Improved Capacitor

It was stated in the Seventh Quarterly Progress Report that a slight change in the C67 composition was made which has resulted in improved life test stability of the Case Size I Monolythic capacitors. The improvement in life test stability indicates an increase in time-to-failure at high temperatures by perhaps a factor of 10. Figure 1 shows typical resistance vs. time curves at 150°C for the obsolete C67 Case Size I Monolythic capacitor and for the improved capacitor. The composition changes leading to increased life test stability have affected neither the dielectric constant nor the stability of dielectric constant with temperature.

Some of the characteristics of the improved capacitor were reported in the Seventh Quarterly Progress Report. Additional characteristics of the improved capacitor were investigated during the eighth quarter, in particular the response of the capacitor to direct voltage as a function of time, temperature, and age.

Curves showing charge current as a function of time at 85°C for a new capacitor at several voltages are presented in Figure 2. The time to reach the condition of steady-state current with a particular voltage at

85°C is about an order of magnitude greater than at 150°C (see Figure 11, Seventh Quarterly Progress Report). All the data presented in Figure 2 were obtained on the same capacitor. The capacitor was charged in steps of increasing voltage, and between chargings, it was stored at 85°C for at least 16 hr. The data in Figure 2 are plotted as steady-state current vs. time in Figure 3. It is seen that current at 85°C is proportional to $V^{1.41}$ below 300 V, which is the same relationship as was found at 150°C (see Figure 12, Seventh Quarterly Progress Report). At 85°C and above, 300 V steady-state current is proportional to $V^{2.60}$.

The data presented so far have been on new capacitors, i.e., on capacitors not previously voltage stressed to any appreciable degree. In Figure 4, data on current as a function of time at 85°C are presented for a capacitor which had previously been subjected to 240 V/mil at 150°C for several hundred hours. The behavior is qualitatively the same as for the new capacitor described in Figure 2. The relationships of steady-state current to applied field at 85°C and 150°C for previously aged capacitors are presented in Figures 5 and 6. As with new capacitors, the conductivity of the aged capacitors is non-ohmic.

The activation energy for conduction of the improved capacitor had previously been determined at 93 V to be 1.05 eV. On the basis of the data shown in Figure 7, the steady-state resistance as a function of temperature at 200 V is plotted in Figure 8. In this instance, the activation energy for conduction of the improved capacitor is 1.00 eV. Because of the scatter in data points, the difference between activation energies at 93 V and 200 V is probably not significant.

Charging current as a function of time at 95 V on an aged capacitor is presented for a variety of temperatures in Figure 9. The steady-state resistance as a function of temperature for an aged capacitor at 95 V and 195 V is plotted in Figure 10. It can be seen on this figure that the activation energy is 1.04 eV. Therefore, the activation energy for conduction of an aged capacitor appears not to be significantly different from that of a new capacitor.

The behavior of the resistance with time of several improved capacitors at 150°C and at several voltages is presented in Figures 11 through 13. The capacitors display stable resistances for 1000 hr or more when stressed as high as 240 V/mil. The capacitors are still on test.

Experimental results presented in the First and Second Quarterly Progress Reports indicated that the mean-time-to-failure on life test of the obsoleted capacitors was somewhat dependent upon the sense of pre-life test direct voltage application. If the sense of the voltage application on life test was the same as before life test, then mean-time-to-failure was less than for capacitors not stressed before life test. However, if the

sense of voltage application before life test was opposite that during life test, mean life times longer than normal were observed. It appears, on the basis of the data presented in Figures 14 and 15, that the improved variety capacitors would display longer life times if, after a period of service, the sense of the voltage could be reversed. These data suggest that it should be possible to examine capacitors at some accelerated condition of temperature and voltage for stability of resistance with time, and, after examination has been made, reverse the voltage for a period of time so as to rejuvenate the capacitors to their initial state. If it could be known positively that a given capacitor had at least a minimum measure of stability at severe conditions of voltage and temperature, then it should be possible to estimate the stability of the capacitor at milder temperature and voltage conditions. The estimation would be based on certain temperature-voltage-time dependencies observed concerning certain characteristics of the obsoleted capacitor.

4.3 Large-Scale Life Testing

A total of 753 Case Size I C67 Monolythic capacitors are now undergoing life test. These capacitors are the original version of this unit, and therefore lifetimes which are shorter than those for the improved version will be observed at a given temperature and voltage. Although the original version is now obsolete, life testing of the 753 capacitors was continued in an attempt to gain knowledge about the lifetime of barium titanate ceramic capacitors.

The capacitors which are on test are grouped as follows:

Class I : This group consists of 200 capacitors which were flashed in the unfired state at 1.5 VDC. A total of 100 of these capacitors were then burned-in at 50 VDC and 150°C for 24 hours, while the other 100 were not. Average capacitance for Class I capacitors is 0.0075 μf.

Class II : This group consists of 293 capacitors which were flashed in the unfired state at 500 VDC. A total of 147 of these capacitors were then burned-in at 50 VDC and 150°C for 24 hours, while the other 146 were not. Average capacitance for Class II units is 0.0085 μf.

Class III : This group consists of 260 capacitors which were flashed in the unfired state at 1000 VDC. A total of 130 of these capacitors were then burned-in at 50 VDC and 150°C for 24 hours, while the other 130 were not. Average capacitance for Class III units is 0.0084 μf.

The purpose of the voltage testing in the unfired state was to eliminate from further processing those capacitors whose counter electrodes are in contact or are separated by gas inclusions or ceramic material having less than nominal thickness. The underlying assumption was that capacitors which displayed such defects before firing would also display defects against long life after firing.

The burn-in conditions of 150°C and 50 V for 24 hr were chosen so that the majority of the capacitors would not be degraded, i.e., have their resistance lowered substantially. Only capacitors which might be early failures in service would be degraded. It was thought that short-lived and long-lived capacitors might be more easily distinguished on the basis of a resistance measurement if the capacitors were subjected to DC voltage for a time before being placed on life test.

Pre-life test measurements of these capacitors before and after burn-in included capacitance and dissipation factor at 1 kc/sec and resistance at 100 VDC and 25°C and at 100 VDC and 150°C after 30 min of electrification. The 30-minute electrification time was chosen because a long time is usually required to reach the point of steady-state resistance. Capacitors which require the longest time to reach this point also appear, in general, to display the longest life times and the highest values of resistance. Therefore, the differences in resistance which will allow differentiation between potentially long-life and short-life capacitors should be more pronounced after 30 minutes of electrification than after only one or two minutes. A review of the background material leading to the approximate life time formula presented in the Sixth and Seventh Quarterly Progress Reports for the original version of this capacitor will help the understanding of this point.

The distribution of resistance values at 25°C and 150°C for the three classes of capacitors was presented in the Sixth Quarterly Progress Report. The distribution at 25°C for each class of capacitors indicated a fairly uniform quality, but the distribution of resistance values at 150°C indicated a variable quality. This variability was greatest for the Class I capacitors. Quality variability among the various classes could not be related to voltage flashing of the unfired parts.

An inspection of the distribution of resistances at 150°C indicated that any capacitor having a resistance greater than 200,000 MΩ could be called normal, while a capacitor having a resistance less than 200,000 MΩ could be considered abnormal or substandard.

The life testing of the units is following a step-stress plan, which to date has comprised the following:

85°C and 50 VDC for 1000 hr; resistance read at 24, 500, and 1000 hr.

85°C and 100 VDC for 500 hr; resistance read at 24, 65, and 500 hr.

105°C and 100 VDC for 1000 hr; resistance read at 500 and 1000 hr.

125°C and 100 VDC for 2000 hr; resistance read at 72, 250, 500, 1000, 1500, and 2000 hr.

The capacitors are continuing on test at 125°C and 100 VDC. The percentage of failures noted at the different readout times for the three classes of capacitors is presented in Table 1. A capacitor is considered a failure when its resistance at test conditions drops below 100 MΩ. This definition is somewhat arbitrary, but it serves to indicate when a normal capacitor is wearing out.

Table 1 indicates a significant difference in mean life times between the three classes of capacitors. Whatever the explanation, this difference cannot, with any confidence, be ascribed to defects in the unfired strips, as detected by flash voltage testing, for the same reasons that the variability of initial resistance at 150°C cannot be explained in this way. These reasons were presented in the Sixth Quarterly Progress Report.

It will be seen on perusal of the data in Table 1 that the percentage of failures actually decreases in several instances as time on test is increased. This occurs because a capacitor may be technically a failure at one readout time, according to the definition adopted, but the resistance may subsequently increase so as to be outside the range of the failure definition. Table 2 in the Seventh Quarterly Progress Report presents many such examples.

Further classification of early failures is presented in Table 2, which has been reproduced from the Seventh Quarterly Progress Report. The classification depends on whether the initial resistance at 150°C after 30 min of electrification with 100 VDC was above 200,000 MΩ, and whether the capacitors were burned-in before the beginning of life testing. The life testing to which the capacitors were subjected is comprised of the 85°C and 105°C voltage conditions and the times shown in Table 1. In compiling data for Table 2, only those capacitors which had resistances greater than 1,000,000 MΩ and 100,000 MΩ at every readout time were included. The same is true for the failures indicated, i.e., if the resistance of a capacitor was less than 100 MΩ at any readout time, it was considered a failure for all subsequent readout times, even if its resistance subsequently increased to a value above the failure point.

TABLE 1

PERCENTAGE OF FAILURES NOTED AT VARIOUS READOUT TIMES

Test Conditions	Readout Time (Hr)	Class I Failures		Class II Failures		Class III Failures	
		Percentage Of Units Burned-In	Percentage Of Units Not Burned-In	Percentage Of Units Burned-In	Percentage Of Units Not Burned-In	Percentage Of Units Burned-In	Percentage Of Units Not Burned-In
85°C, 50 VDC	24	0.0	0.0	0.7	2.1	1.6	0.8
85°C, 50 VDC	500	2.0	1.0	0.0	3.4	0.8	0.8
85°C, 50 VDC	1000	1.0	0.0	0.7	2.8	0.8	0.0
85°C, 100 VDC	24	2.0	1.0	2.1	3.4	0.8	0.0
85°C, 100 VDC	65	2.0	1.0	2.1	2.1	0.8	0.0
85°C, 100 VDC	500	4.0	1.0	0.7	2.1	0.8	0.0
105°C, 100 VDC	500	3.0	2.0	2.7	3.4	0.8	0.0
105°C, 100 VDC	1000	3.0	2.0	3.4	4.7	1.6	0.0
125°C, 100 VDC	72	8.0	10.0	12.2	8.2	2.3	1.6
125°C, 100 VDC	250	19.0	14.0	17.0	13.5	3.1	3.1
125°C, 100 VDC	500	27.0	18.0	12.2	16.4	2.3	3.1
125°C, 100 VDC	1000	41.0	30.0	25.8	22.6	7.0	12.3
125°C, 100 VDC	1500	69.0	69.0	43.5	40.5	13.9	19.2
125°C, 100 VDC	2000	81.0	83.0	47.0	47.3	13.9	21.5

Failure defined as resistance less than 100 MΩ at test conditions.

TABLE 2

COMPARISON OF LIFE TEST PERFORMANCE OF CAPACITORS HAVING 150°C RESISTANCE ABOVE AND BELOW 200,000 MΩ

Class	Burn-In Yes	Burn-In No	No. of Units With Resistance >200,000 MΩ At 150°C	No. of Units With Resistance <200,000 MΩ At 150°C	Percentage of Units Having Resistance On Life Test >1,000,000 MΩ	Percentage of Units Having Resistance On Life Test >100,000 MΩ	Percentag Of Failure
I		x		91	9	50	5.5
I		x	9		44	89	0.0
I	x		8		63	75	0.0
I	x			92	9	37	4.4
II		x		62	0	37	13.0
II		x	84		23	81	2.4
II	x		51		22	90	2.0
II	x			96	1	29	8.3
III		x		31	10	52	3.2
III		x	99		19	64	1.0
III	x		93		37	96	1.1
III	x			37	8	70	5.4

Notes: (1) Life Test Schedule: 85°C, 50 VDC, 1000 hr; readouts at 24 hr, 500 hr, 1000 hr.
85°C, 100 VDC, 500 hr; readouts at 24 hr, 65 hr, 500 hr.
105°C, 100 VDC, 1000 hr; readouts at 500 hr, 1000 hr

(2) Failure defined as resistance less than 100 MΩ at test conditions.

(3) For capacitors which received burn-in, initial resistance refers to the resistance value after burn-in.

The classification point of 200,000 MΩ initial resistance at 150°C was chosen because earlier inspection of resistance distributions seemed to indicate that capacitors having resistances greater than this value might be considered normal, while those showing lower resistances might be considered abnormal.

The conclusion reached regarding the information in Table 2 is that capacitors having initial resistance at 150°C greater than 200,000 MΩ will produce a smaller percentage of early failures on life test than capacitors having initial resistance less than 200,000 MΩ. This conclusion seems valid whether or not a capacitor is defined a failure when its resistance at test conditions becomes less than 1,000,000 MΩ, less than 100,000 MΩ, or less than 100 MΩ. It is more difficult to reach a conclusion regarding a comparison between capacitors having initial resistance greater than 200,000 MΩ after burn-in and capacitors not burned-in but having initial resistance greater than 200,000 MΩ. In general, it appears that capacitors will be more reliable, with respect to early failure, if they are burned-in before measurement of initial resistance than if they are not burned-in. This conclusion is clearly demonstrated in the case of the Class III capacitors (and, to some extent, in the case of the other two classes) at the resistance divisions of 1,000,000 MΩ and 100,000 MΩ.

The analysis of the life test data on Class III capacitors has been extended to include testing at 125°C and 100 VDC for 1000 hr, in addition to the preceding schedule of life testing at 85°C and 105°C. The data on Class I and Class II capacitors were not analyzed beyond the conditions and times at 105°C because these classes demonstrate a sizeable percentage of failures after only a short time at 125°C, as can be seen in Table 1. An extended analysis might therefore tend to confuse the relationship between early failures, burn-in, and initial resistance.

The extended analysis of life test data for Class III capacitors is presented in Table 3. The most reliable capacitors are those which received burn-in and which after burn-in had resistances exceeding 200,000 MΩ at 150°C after 30 min electrification with 100 VDC. This statement is clearly true if failure is taken to mean a resistance less than 1,000,000 MΩ, or less than 100,000 MΩ, or less than 10,000 MΩ at test conditions.

It is not surprising that capacitors having high initial resistance at 150°C (i.e., greater than 200,000 MΩ) should, in general, display greater reliability than capacitors having low resistance at 150°C. The life time formula for individual capacitors described in the Seventh Quarterly Progress Report contains two important elements relating directly to life time, namely, time-to-peak-resistance and peak resistance. In the majority of instances, these are related to initial resistance as it is being measured here. In Figure 2 of the Sixth Quarterly Progress Report, it can be seen that high values of resistance at 150°C after several minutes of charging seem to indicate high values of peak resistance. In Figure 9 of the Seventh Quarterly Progress

TABLE 3

COMPARISON OF LIFE TEST PERFORMANCE OF CLASS III CAPACITORS HAVING INITIAL 150°C RESISTANCE ABOVE AND BELOW 200,000 MΩ

Burn-In Yes	Burn-In No	No. of Units With Initial Resistance >200,000 MΩ At 150°C	No. of Units With Initial Resistance <200,000 MΩ At 150°C	Percentage of Units Having Resistance on Life Test >1,000,000 MΩ	Percentage of Units Having Resistance on Life Test >100,000 MΩ	Percentage of Units Having Resistance on Life Test >10,000 MΩ	Percentage Of Failures
	x		31	3.2	22.5	32.2	22.5
	x	99		8.1	43.3	55.6	10.1
x		93		22.6	66.7	81.7	7.5
x			37	8.1	29.7	59.5	8.1

Notes: (1) Life Test Schedule: 85°C, 50 VDC, 1000 hr; readouts at 24, 500, and 1000 hr
85°C, 100 VDC, 500 hr; readouts at 24, 65, and 500 hr
105°C, 100 VDC, 1000 hr; readouts at 500, 1000 hr
125°C, 100 VDC, 1000 hr; readouts at 72, 250, 500, and 1000 hr

(2) Failure defined as resistance less than 100 MΩ at test conditions.

(3) For capacitors which were burned-in, initial resistance refers to resistance value after burn-in.

Report, it can be seen that capacitors displaying long times to peak resistance generally have the highest values of peak resistance. Therefore, in the majority of instances, it can be expected that, after several minutes of charging, capacitors displaying normal or high values of resistance relative to the population as a whole at accelerated conditions of testing will be more reliable with respect to the occurrence of early failures on life test than those capacitors displaying abnormally low values of initial resistance.

4.4 Life Time of C67 Case Size I Monolythic Capacitors

The Seventh Quarterly Progress Report presented the details of an approximate life time formula derived on the basis of the resistance vs. time features of the obsoleted version of this capacitor at various temperature and voltage conditions. The principal elements in this formula were peak resistance, time-to-peak-resistance, and a degradation rate. The principal elements can be readily discerned from the resistance vs. time curve for an obsoleted capacitor presented in Figure 1 of this report.

An experiment to determine if an empirical relationship exists between time-to-peak-resistance or peak resistance at 150°C and 185 VDC and life test behavior at lower temperatures for 27 C67 Case Size I Monolythic capacitors was begun several months ago and has now been terminated. The capacitors used in the experiment ranged in capacity from 6,000 to 10,000 μμf and were from three different lots.

The life testing of these capacitors followed a step-stress design according to the following schedule:

85°C and 100 V for 340 hr
85°C and 200 V for 1500 hr
105°C and 200 V for 2350 hr
125°C and 200 V for 524 hr

The resistance of the capacitors was periodically determined at test conditions. The characteristics of these capacitors, such as time-to-peak-resistance and peak resistance as well as the resistance of the capacitors at various times during testing, are presented in Table 4.

Not all the capacitors were severely degraded during the course of testing. None of the capacitors failed catastrophically. Because of the test circuitry, the applied voltage was divided between the test capacitor and a 1 MΩ resistor in series with the capacitor. Therefore, when the resistance of the capacitor on test dropped to less than 10 MΩ, a substantial fraction of the applied voltage was removed from the test capacitor.

TABLE 4

LIFE TEST PERFORMANCE OF CASE SIZE I C67 MONOLYTHIC CAPACITORS AT 85°C, 105°C, 125°C

Unit No.	t_p (Min.) At 85 VDC 150°C	R_p (MΩ) At 185 VDC 150°C	100 VDC, 85°C MΩ at 24 Hr	100 VDC, 85°C MΩ at 340 Hr	200 VDC, 85°C MΩ at 600 Hr	200 VDC, 85°C MΩ at 1500 Hr	200 VDC, 105°C MΩ at 24 Hr	200 VDC, 105°C MΩ at 1300 Hr	200 VDC, 105°C MΩ at 2350 Hr	200 VDC, 125°C MΩ at 22 Hr	200 VDC, 125°C MΩ at 210 Hr	200 VDC, 125°C MΩ at 524 Hr
583-16	1.3	18,200	>330,000	>330,000	125,000	21,500	5,880	23	11	6	3	2
583-17	8.5	38,100	>330,000	>330,000	>660,000	182,000	50,000	52	11	6	2	1
583-18	11	127,000	>330,000	>330,000	>660,000	>660,000	445,000	29,400	2,980	560	1,290	425
583-341	270	1,880,000	>330,000	>330,000	>660,000	>660,000	>660,000	>660,000	>660,000	69.000	20,000	4,000
583-24	120	1,030,000	>330,000	>330,000	>660,000	>660,000	>660,000	4,770	3,110	1,600	1,430	690
583-26	28	555,000	>330,000	>330,000	573,000	286,000	125,000	181	33	16	10	3
583-27	344	1,100,000	>330,000	>330,000	>660,000	>660,000	>660,000	>660,000	440,000	74,000	11,000	5,000
583-28	15	580	>330,000	>330,000	>660,000	286,000	33,000	64	40	13	8	2
583-29	82	844,000	>330,000	>330,000	>660,000	>660,000	>660,000	286,000	360	150	8	2
583-30	300	1,030,000	>330,000	>330,000	>660,000	>660,000	>660,000	500,000	156,000	26,000	9,500	2,940
583-31	240	1,290,000	>330,000	>330,000	>660,000	>660,000	>660,000	4,000	330	142	110	24
583-32	268	1,440,000	>330,000	>330,000	>660,000	>660,000	>660,000	11,100	1,660	835	645	475
583-33	105	1,030,000	>330,000	>330,000	45,000	3,640	2,540	95	82	62	52	62
583-34	22	197,000	>330,000	>330,000	>660,000	>660,000	>660,000	77,000	6,650	735	117	9
583-35	390	1,630,000	>330,000	>330,000	>660,000	>660,000	>660,000	>660,000	>660,000	>660,000	>660,000	>660,000
427-11	330	3,000,000	>330,000	>330,000	>660,000	>660,000	>660,000	>660,000	>660,000	250,000	143,000	66,000
427-12	270	2,420,000	>330,000	>330,000	>660,000	>660,000	>660,000	>660,000	>660,000	500,000	250,000	84,000
427-15	120	1,830,000	>330,000	>330,000	>660,000	>660,000	>660,000	500,000	250,000	65,000	37,000	14,000
427-16	480	3,320,000	>330,000	>330,000	>660,000	>660,000	>660,000	>660,000	>660,000	400,000	400,000	105,000
427-74	330	3,510,000	>330,000	>330,000	>660,000	>660,000	>660,000	>660,000	>660,000	250,000	220,000	77,000
427-75	346	2,740,000	>330,000	>330,000	>660,000	>660,000	>660,000	>660,000	>660,000	335,000	400,000	221,000
449-1	510	3,330,000	>330,000	>330,000	>660,000	>660,000	>660,000	>660,000	>660,000	>660,000	>660,000	>660,000
449-2	480	3,210,000	>330,000	>330,000	>660,000	>660,000	>660,000	>660,000	>660,000	>660,000	>660,000	>660,000
449-3	480	2,720,000	>330,000	>330,000	>660,000	>660,000	>660,000	>660,000	>660,000	>660,000	>660,000	>660,000
449-4	480	3,380,000	>330,000	>330,000	>660,000	>660,000	>660,000	>660,000	>660,000	>660,000	>660,000	>660,000
449-5	480	3,450,000	>330,000	>330,000	>660,000	>660,000	>660,000	>660,000	>660,000	>660,000	>660,000	>660,000
449-6	480	2,640,000	>330,000	>330,000	>660,000	>660,000	>660,000	>660,000	>660,000	>660,000	>660,000	>660,000

In general, the data in Table 4 indicate a relationship between time-to-peak-resistance and stability of resistance on life test. Capacitors having short times-to-peak-resistance at 150°C and 185 VDC degraded to low values of resistance on life test, whereas capacitors showing long times-to-peak-resistance did not degrade on life test.

In practice, the selection for potentially long life of titanate ceramic capacitors displaying resistance vs. time characteristics similar to the obsoleted version of the C67 Case Size I Monolythic capacitors would be made on the basis of resistance determinations at 150°C or other severe temperatures for a period of time sufficient to ensure that the capacitor has at least a certain minimum value of time-to-peak-resistance.

4.5 Selection of Long Life Capacitors of the Improved Version

An experiment has been started with the goal of establishing a procedure for the selection of long life capacitors of the improved version. The experiment is designed not only to demonstrate that the selection procedure is effective but also to supply evidence to support assumptions made about the improved capacitor. Information previously obtained on the obsoleted version of the C67 Case Size I Monolythic capacitor was used to guide the design of the experiment.

An outline of the different parts of the experiment is presented in Figure 16. Essentially, the plan to demonstrate that the life lost by a capacitor during a brief period of accelerated testing can be regained by application of voltage of opposite sense for a time. That this is true for the obsoleted version of the capacitor was demonstrated in the First and Second Quarterly Reports. Further, it is hoped to demonstrate that if a capacitor is capable of a given performance at some accelerated condition, it will deliver at least a certain minimum standard of performance at milder conditions.

The following two-step screening technique is used in the experiment:

Step 1 - The capacitors are subjected to 300 VDC at 150°C for 72 hr, during which time the resistance of acceptable capacitors may not vary more than 20%.

Step 2 - The acceptable capacitors from Step 1 are subjected to 300 VDC applied in the opposite sense at 150°C for 72 hr, during which time the resistance of acceptable capacitors may not vary more than 20%.

The purpose of Step 1 of the screening program is to identify those capacitors which have not reached the point of onset of degradation. These capacitors would be subjected to Step 2, while those capacitors displaying degradation would be discarded. In this experiment, however, no capacitors will be discarded.

The purpose of Step 2 of the screening program is to rejuvenate normal capacitors and to detect those capacitors which might be sensitive to polarity because of some construction or material defect.

The tolerance of 20% on resistance variation may be reduced if very close control over temperature and electrical interference can be achieved.

If it is assumed that a capacitor which passes the screening program can survive 300 VDC of either sense at 150°C for 72 hr without reaching the onset of degradation, the minimum time-to-onset-of-degradation can then be calculated, making use of relationships derived from the obsoleted version and accommodating them to the improved version with certain other assumptions. In particular, the accommodated relationships used to estimate minimum time-to-onset-of-degradation are:

$$t_d = t_o \exp\left(\frac{\Delta w}{kT}\right) \quad \text{where } \Delta w = 0.90 \text{ eV}$$

$$\text{and } t_d \propto V^{-2.7}$$

Some sample calculated results are:

Conditions	Minimum Time-to-Onset-of-Degradation
150°C and 100 VDC	8 weeks
150°C and 50 VDC	52 weeks
125°C and 200 VDC	6 weeks
125°C and 100 VDC	38 weeks
125°C and 50 VDC	4.8 years
85°C and 200 VDC	2.2 years
85°C and 100 VDC	14 years
85°C and 50 VDC	89 years

A detailed presentation of data obtained to date on the capacitors being used in the experiment outlined in Figure 16 is given in Figures 17 through 24.

The distribution of capacitance values is shown in Figure 17. Average dissipation factor at 1 kc/sec., 0.5 V_{rms}, was 1.0%. The extreme values of dissipation factor were 0.9% and 1.1%.

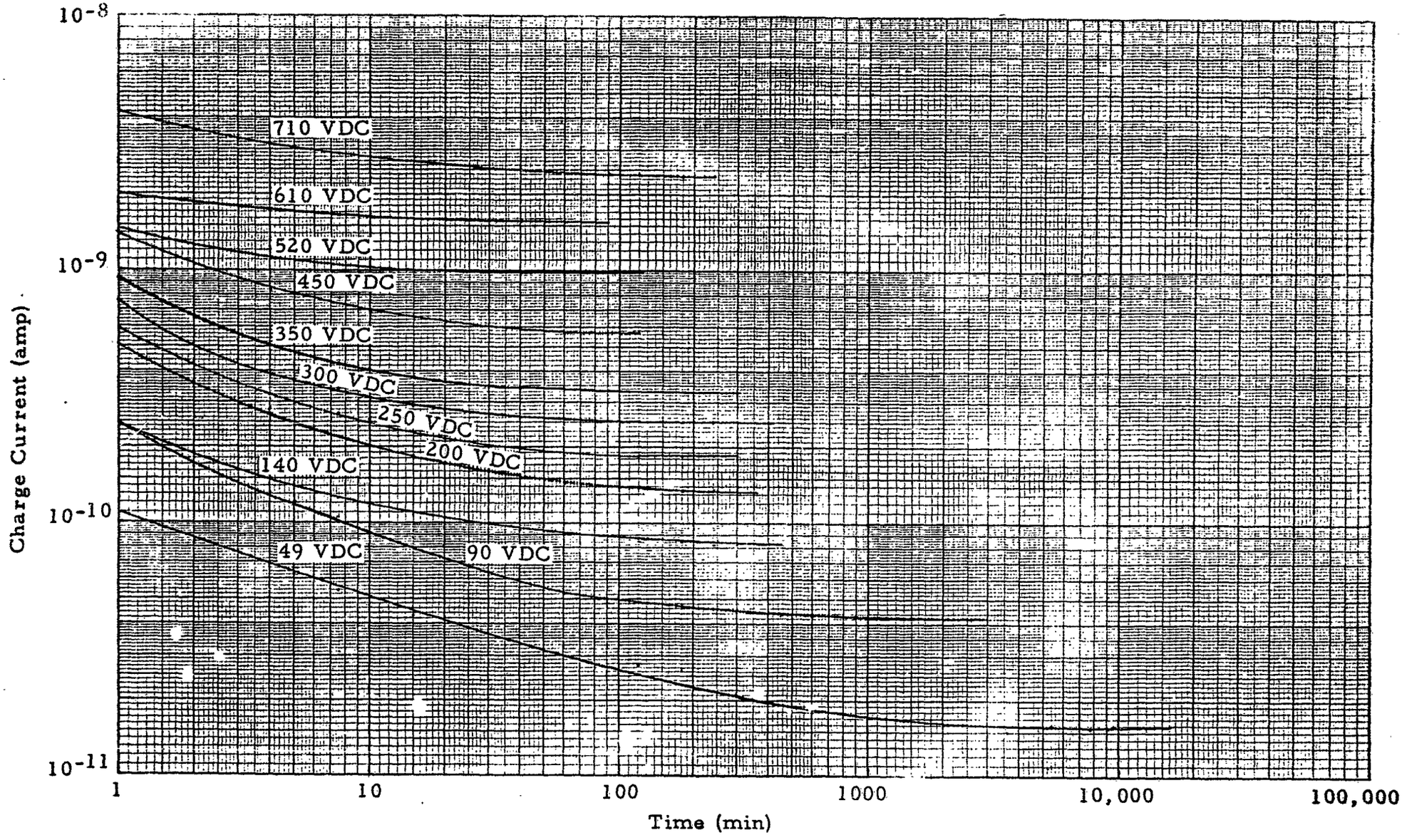
Figure 18 shows the distribution of RC products for the capacitors before the screening program. In calculating RC product resistance at 100 VDC and 150°C, 30±3 min electrification was used together with capacitance at 25°C, 1 kc/sec., 0.5 V_{rms}. The spread in RC products is about one order of magnitude.

Figure 19 shows the distribution of RC products after one hour and after 72 hr in Step 1 of the screening program, and Figure 20 shows the distribution of RC product changes between one hour and 72 hr in the program. During the course of burn-in, the resistance of most capacitors decreases. For two per cent of the capacitors, the resistance decreases more than 20%. Figure 21 shows resistance vs. time during Step 1 of the screening program for an average capacitor (No. 225) and three non-typical capacitors. Except for capacitor No. 246, the non-typical capacitors during Step 1 had typical RC product values before the screening program.

Figure 22 shows the distribution of RC products after one hour and after 72 hr in Step 2 of the screening program. None of the capacitors from Step 1 which behaved unusually (i.e., Nos. 246, 253, and 260) were subjected to Step 2. Figure 23 shows the distribution of RC product changes between one hour and 72 hr in Step 2 of the screening program. There were no unusually behaving capacitors detected in Step 2.

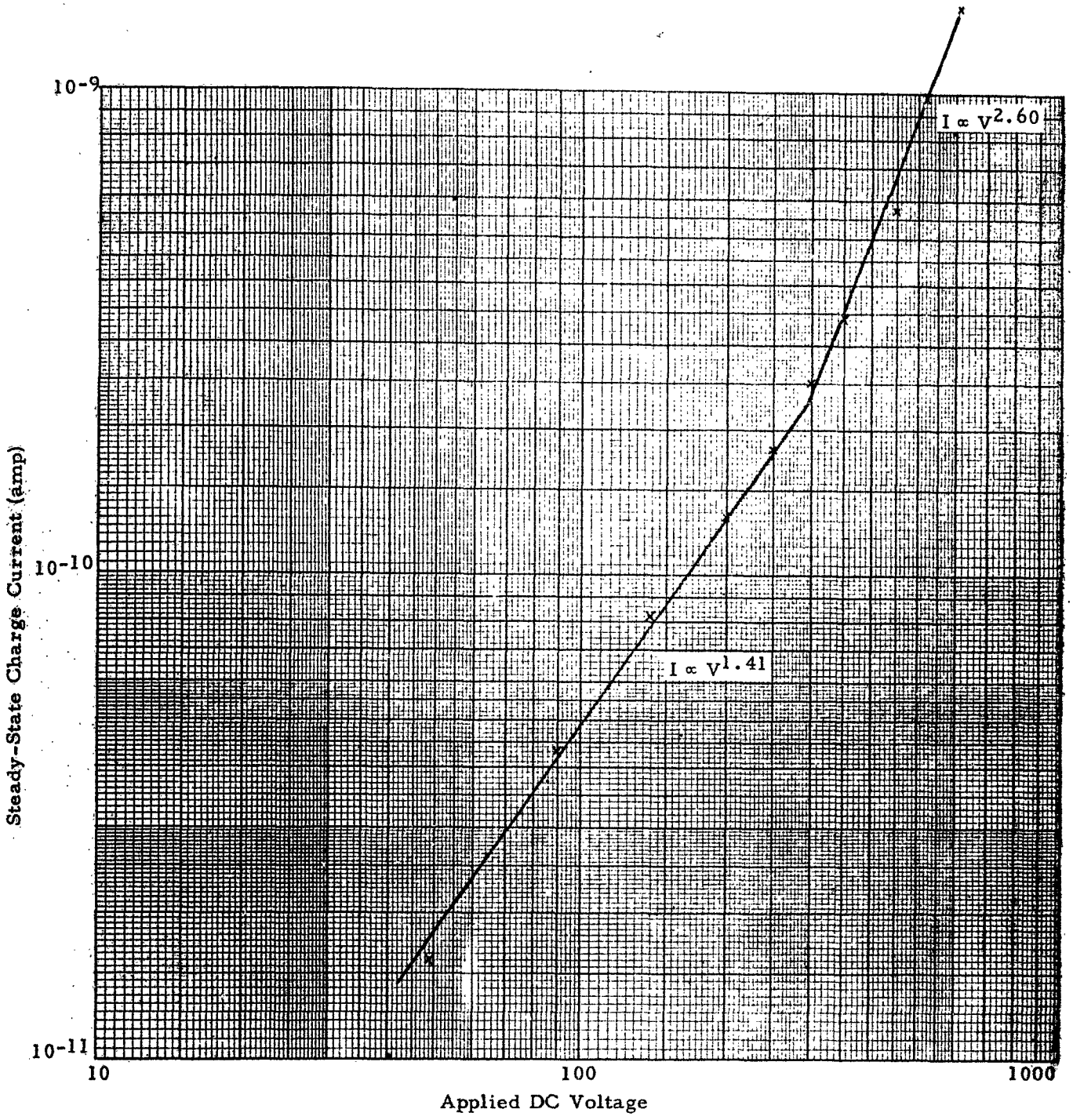
The capacitors outlined in Figure 16 are now on life test at 125°C and 200 VDC. After 250 hr at this condition, none of the capacitors which survived the screening program has reached the onset of degradation. (These capacitors are in positions A and B of Figure 16.) The life test behavior of the average capacitor and the non-typical capacitors described in Figure 21 is shown in Figure 24. Capacitor Nos. 225 and 246 are in position D in the Figure 16 outline, and Capacitor Nos. 253 and 260 are in position C.

Life testing of the capacitors is continuing.



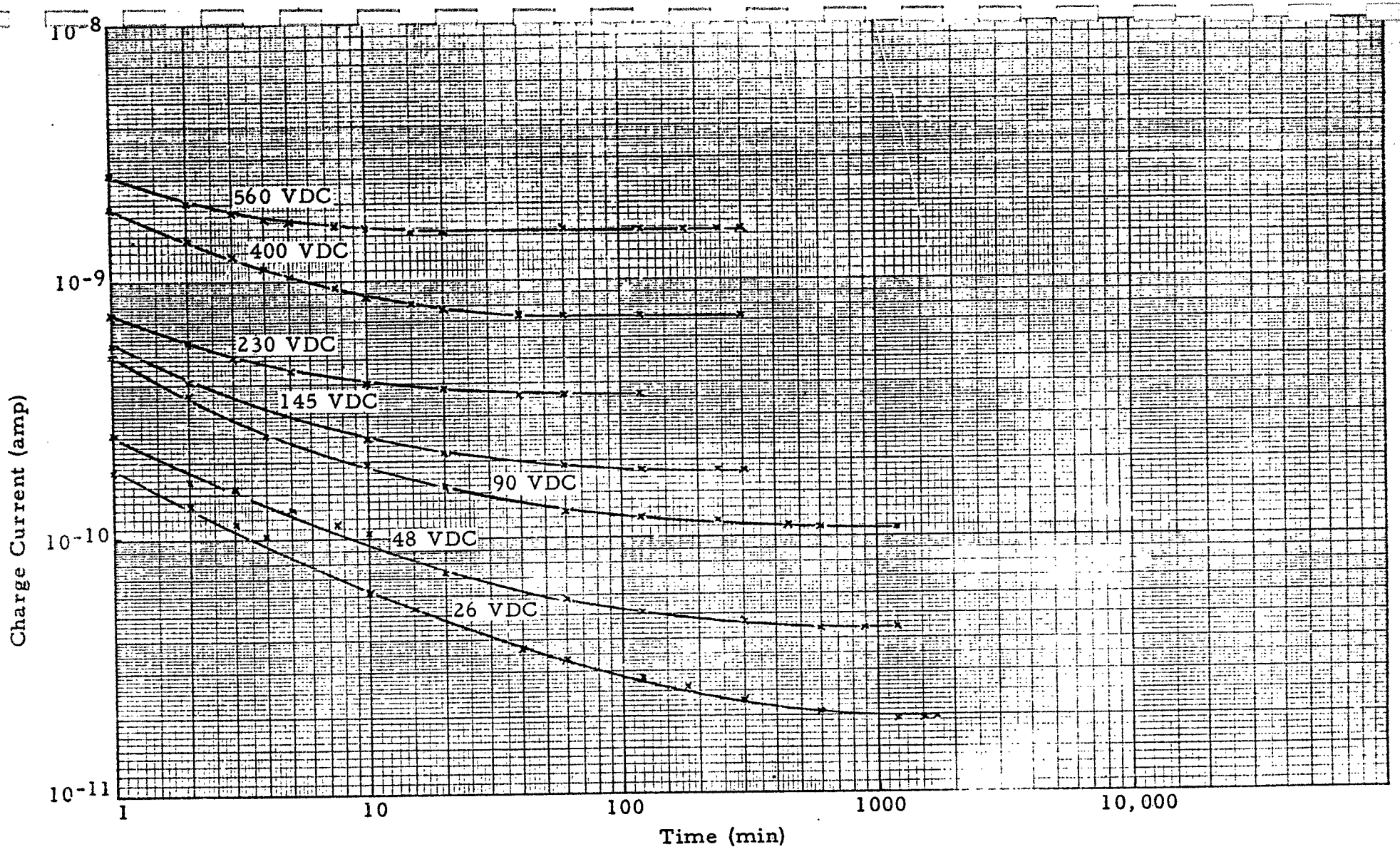
CHARGE CURRENT VS TIME AT 85°C
FOR A NEW, IMPROVED 0.01 μf C67 CASE SIZE I MONOLYTHIC CAPACITOR (Lot 830)
Dielectric Thickness: 0.0025 in.

Figure 2



STEADY-STATE CHARGE CURRENT VS VOLTAGE AT 85°C
FOR A NEW, IMPROVED 0.01 μf C67 CASE SIZE I MONOLYTHIC CAPACITOR
Dielectric Thickness: 0.0025 in.

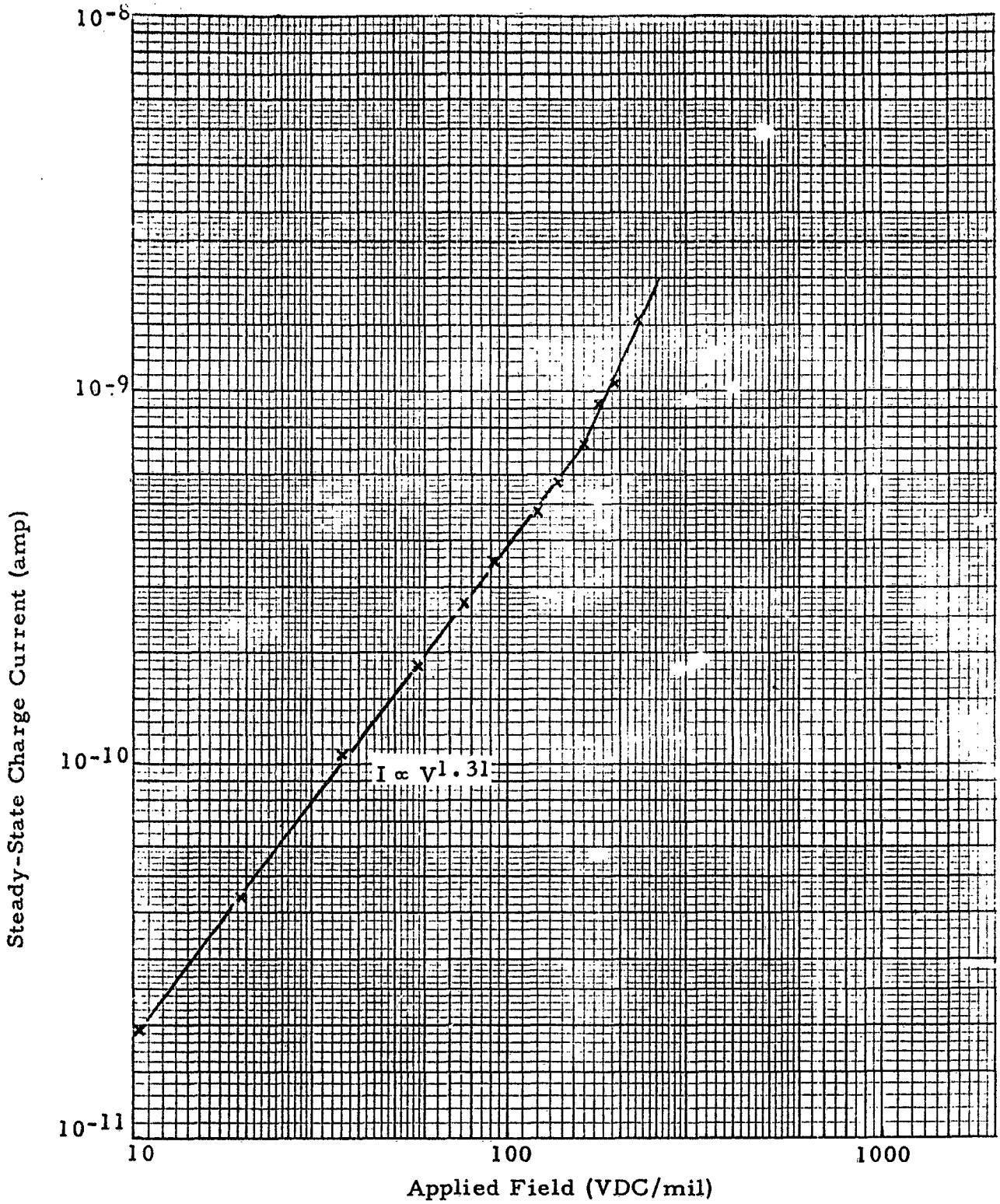
Figure 3



CHARGE CURRENT VS TIME AT 85°C
FOR AN IMPROVED 0.01 μf C67 CASE SIZE I MONOLYTHIC CAPACITOR (Lot 830) WHICH WAS AGED

Notes: (1) Charge current applied in same polarity as used during aging
(2) Capacitor aged at 150°C for 700 hr with 600 VDC.
During aging, resistance decreased from 10,000 MΩ to 700 MΩ

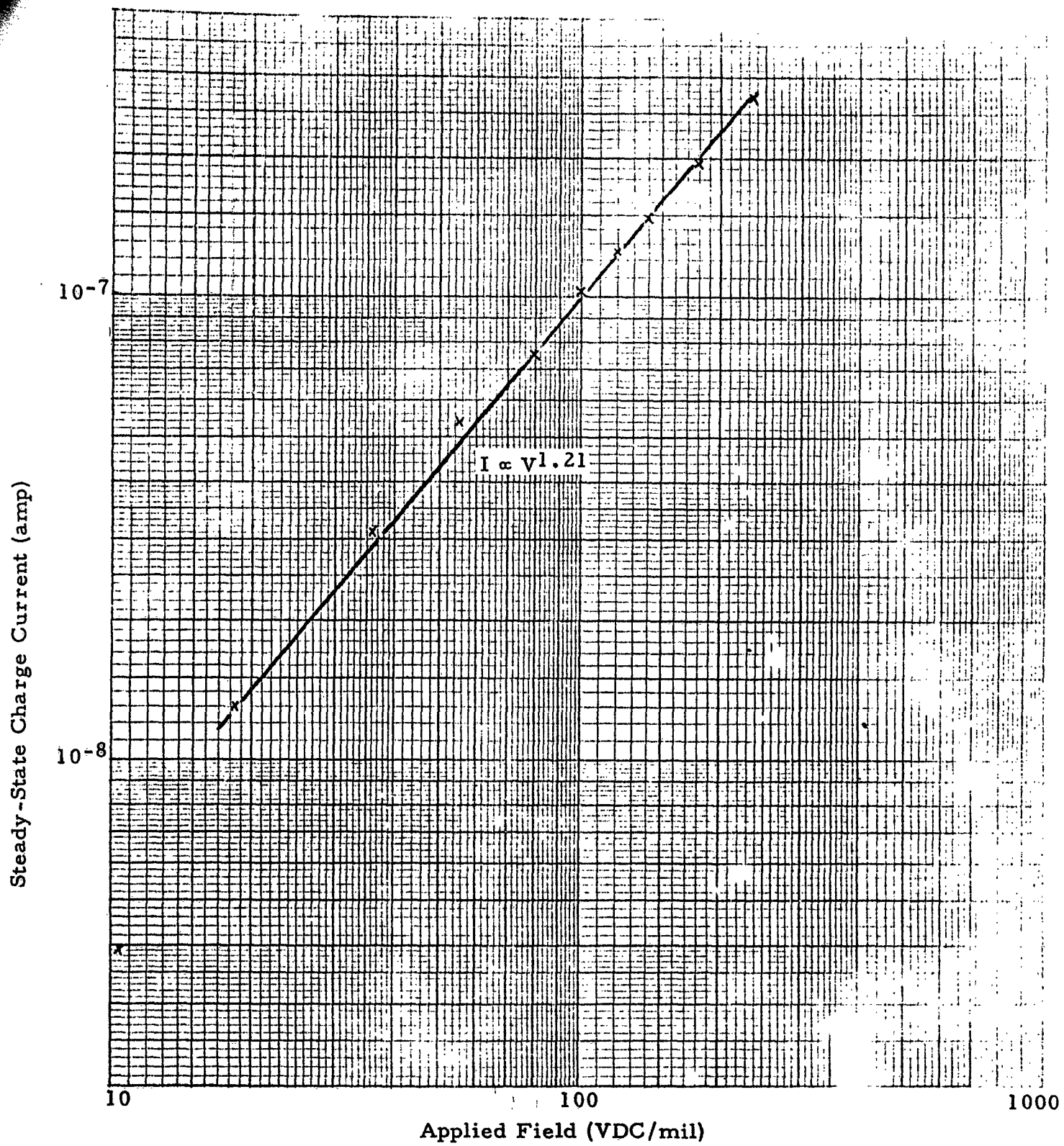
Figure 4



STEADY-STATE CHARGE CURRENT VS APPLIED FIELD AT 85°C
FOR AN IMPROVED 0.01 μf C67 CASE SIZE I
MONOLYTHIC CAPACITOR (Lot 830) WHICH WAS AGED

Notes: (1) Charge current applied in same polarity as used during aging
(2) Capacitor aged at 150°C for 700 hr with 240 VDC/mil.
During aging, resistance decreased from 10,000 MΩ to 700 MΩ

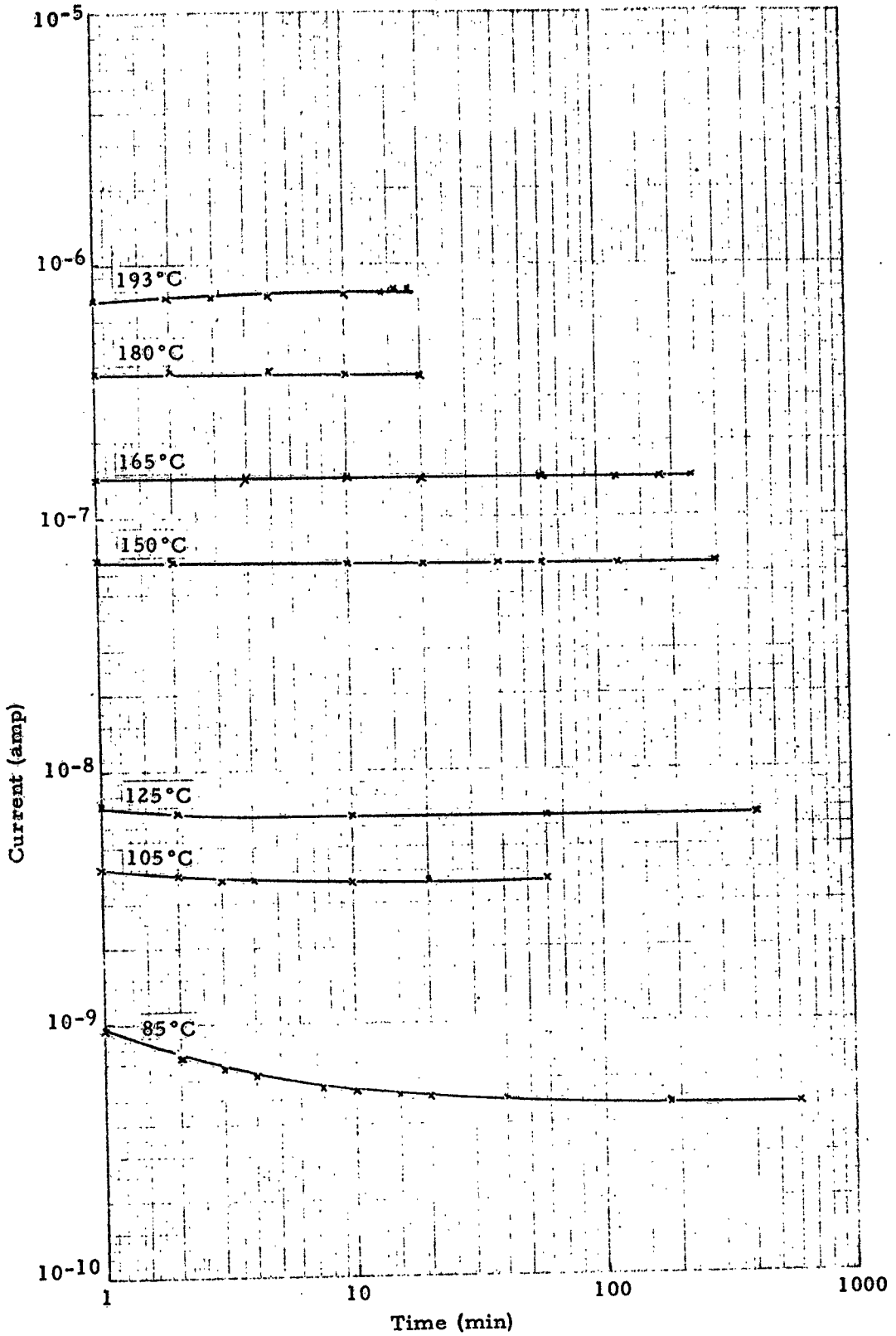
Figure 5



STEADY-STATE CHARGE CURRENT VS APPLIED FIELD AT 150 °
FOR AN IMPROVED 0.01 μf C67 CASE SIZE I
MONOLYTHIC CAPACITOR (Lot 830) WHICH WAS AGED

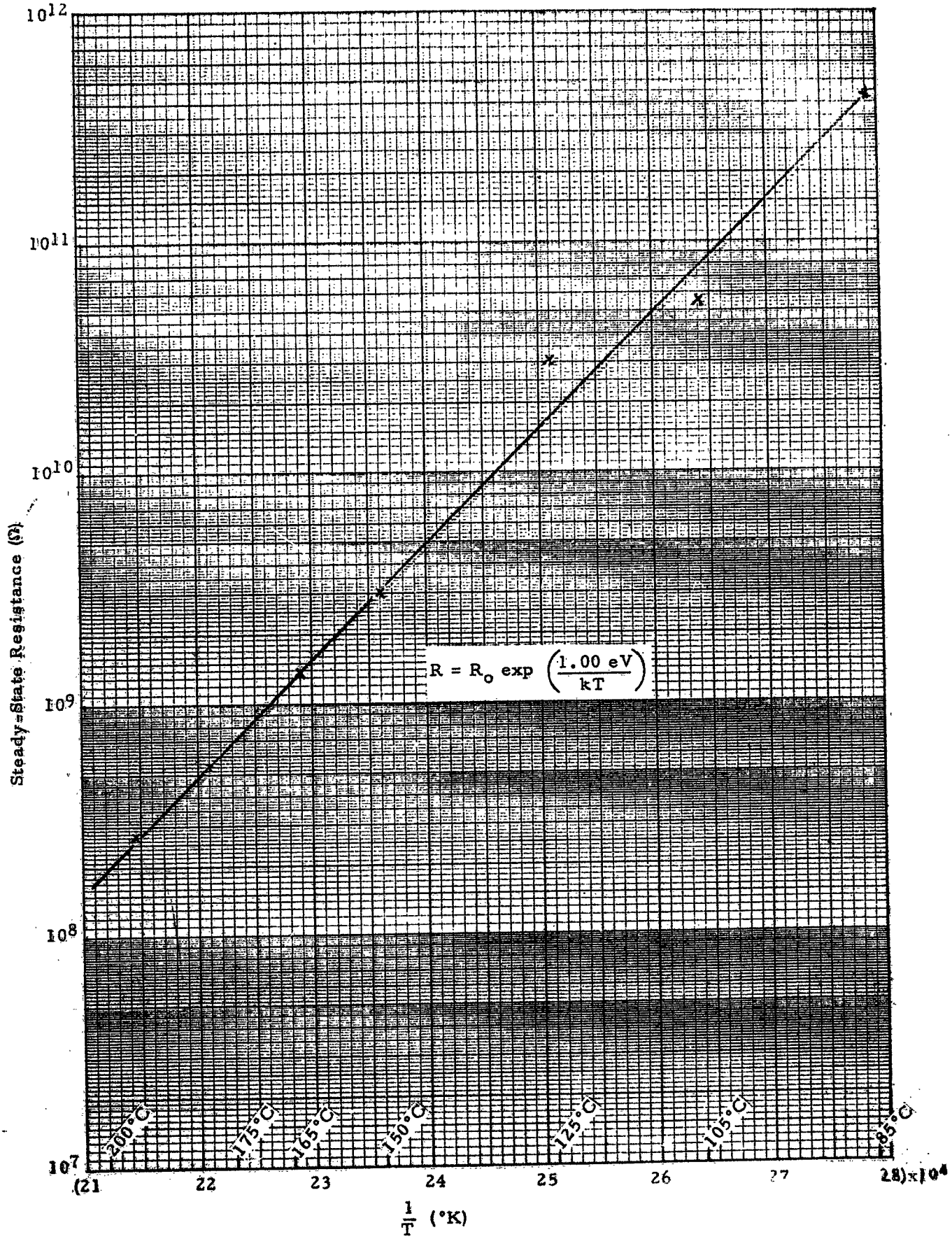
Notes: (1) Charge current applied in same polarity as used during aging
(2) Capacitor aged at 150°C for 820 hr with 240 VDC/mil.
During aging, resistance decreased from 10,400 MΩ to 2400 MΩ

Figure 6

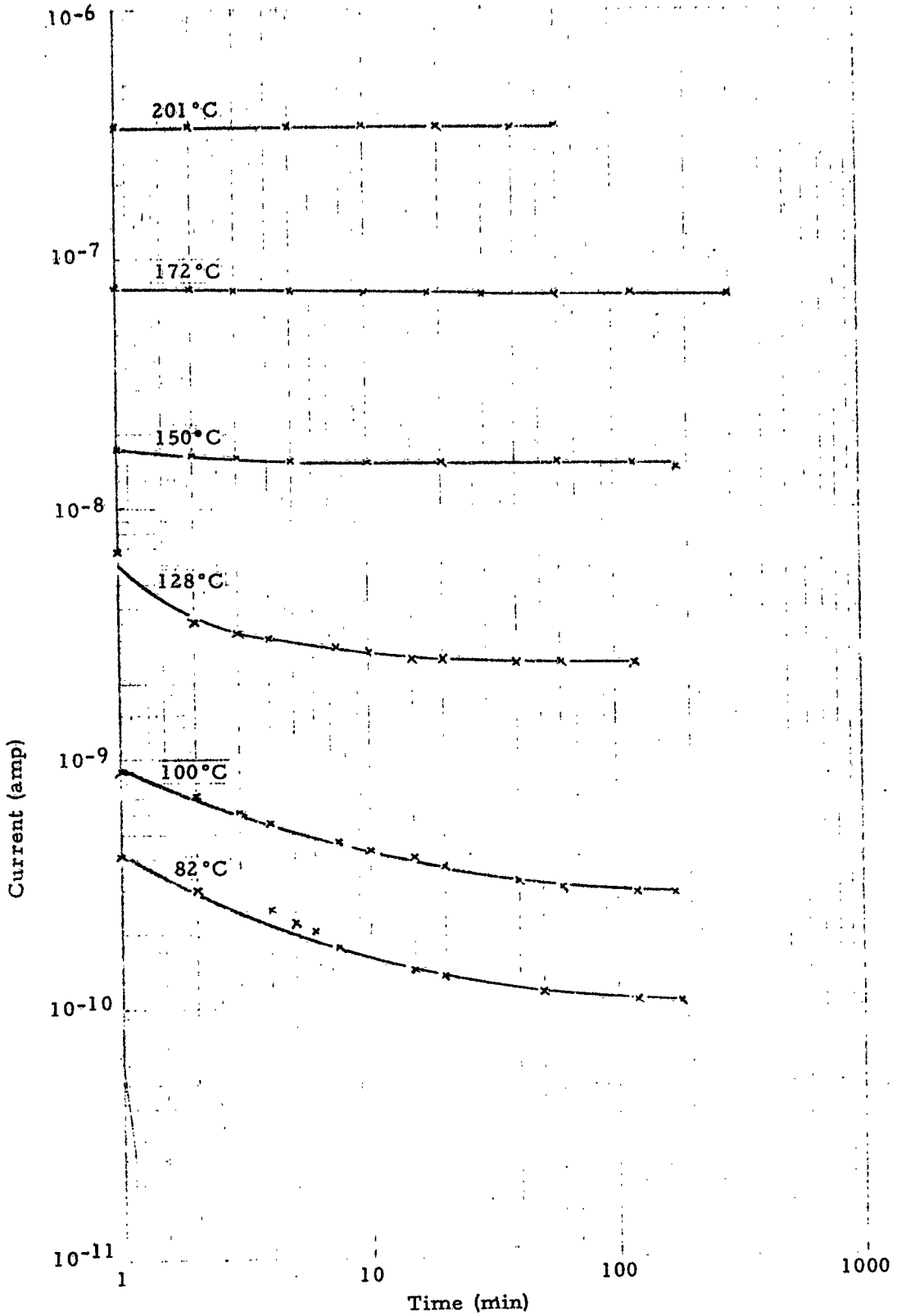


CURRENT VS TIME AT 200 VDC
FOR A NEW, IMPROVED 0.01 μf C67 CASE SIZE I
MONOLYTHIC CAPACITOR (Lot 830)

Figure 7



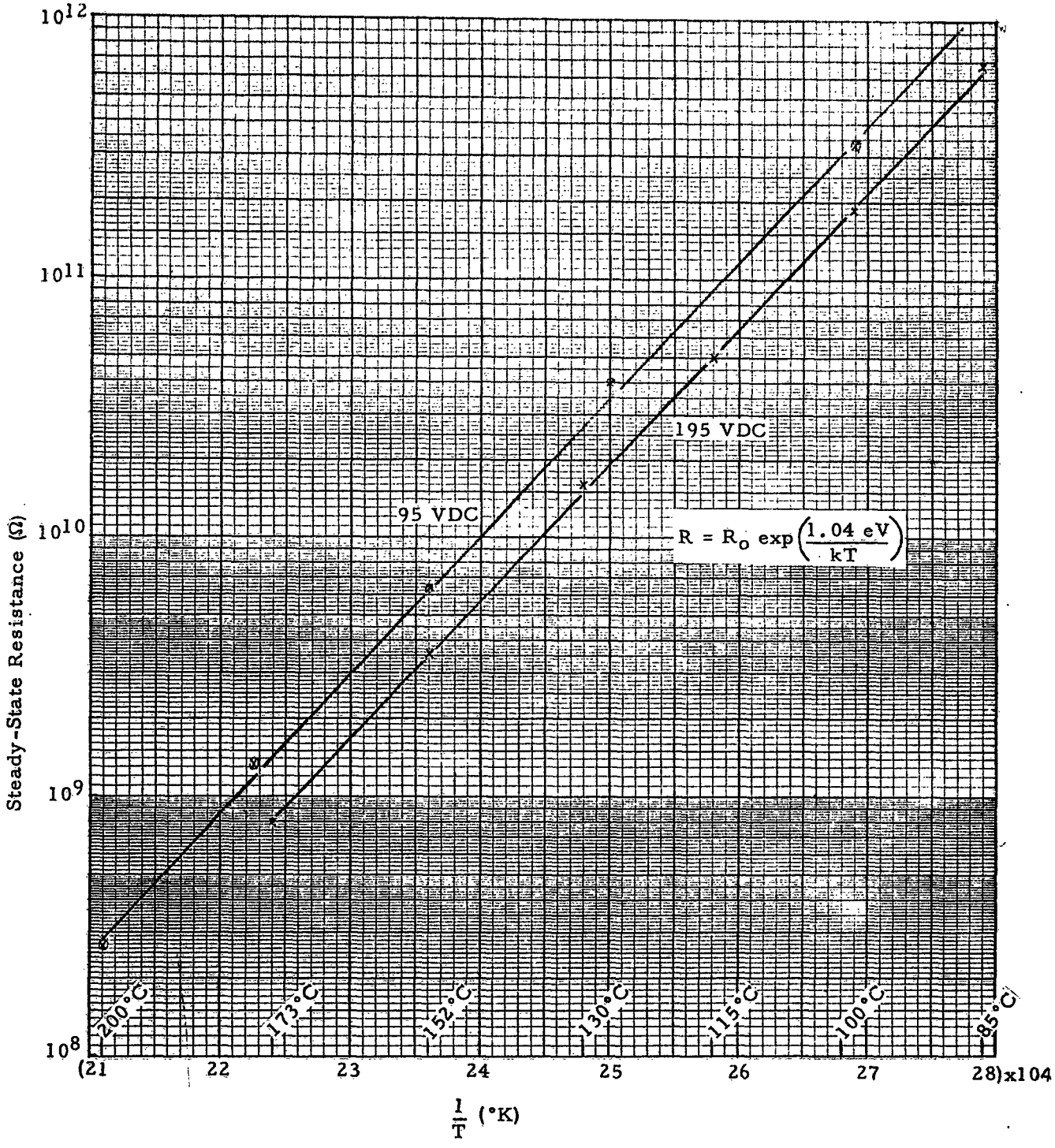
STEADY-STATE RESISTANCE
VS INVERSE ABSOLUTE TEMPERATURE AT 200 VDC
FOR A NEW, IMPROVED 0.01 μf C67 CASE SIZE I MONOLYTHIC CAPACITOR



CURRENT VS TIME AT 95 VDC
FOR AN IMPROVED 0.01 μf C67 CASE SIZE I
MONOLYTHIC CAPACITOR (Lot 830) WHICH WAS AGED

Notes: (1) Charge current applied in same polarity as used during aging
(2) Capacitor aged at 150°C for 700 hr with 600 VDC.
During aging, resistance decreased from 10,000 MΩ to 700 MΩ

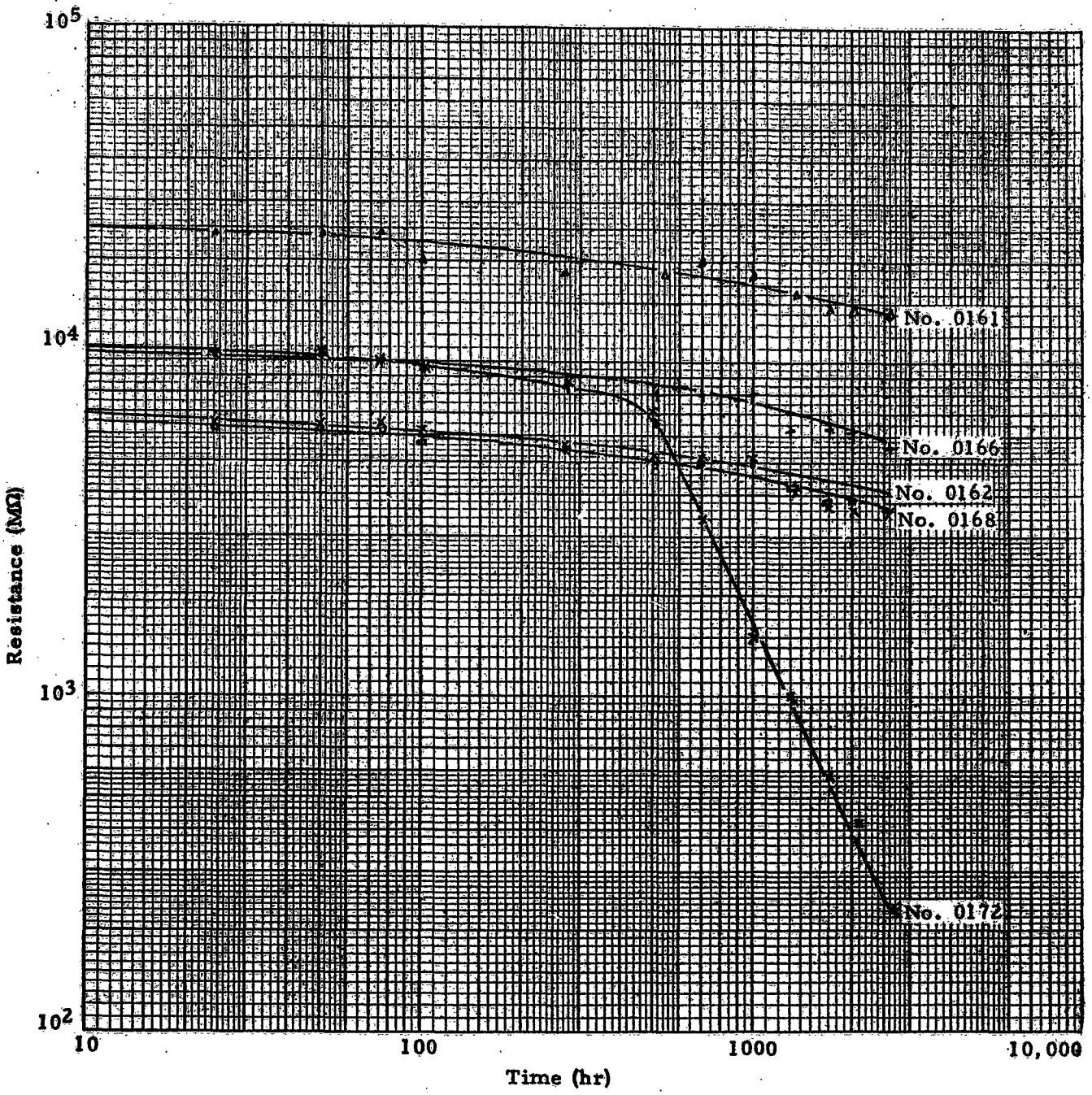
Figure 9



STEADY-STATE RESISTANCE
VS INVERSE ABSOLUTE TEMPERATURE AT 95 VDC AND 195 VDC
FOR AN IMPROVED 0.01 μf C67 CASE SIZE I
MONOLYTHIC CAPACITOR (Lot 830) WHICH WAS AGED
Dielectric Thickness: 0.0025 in.

Note: Capacitor aged at 150°C for 700 hr with 600 VDC.
During aging, resistance decreased from 10,000 MΩ to 700 MΩ

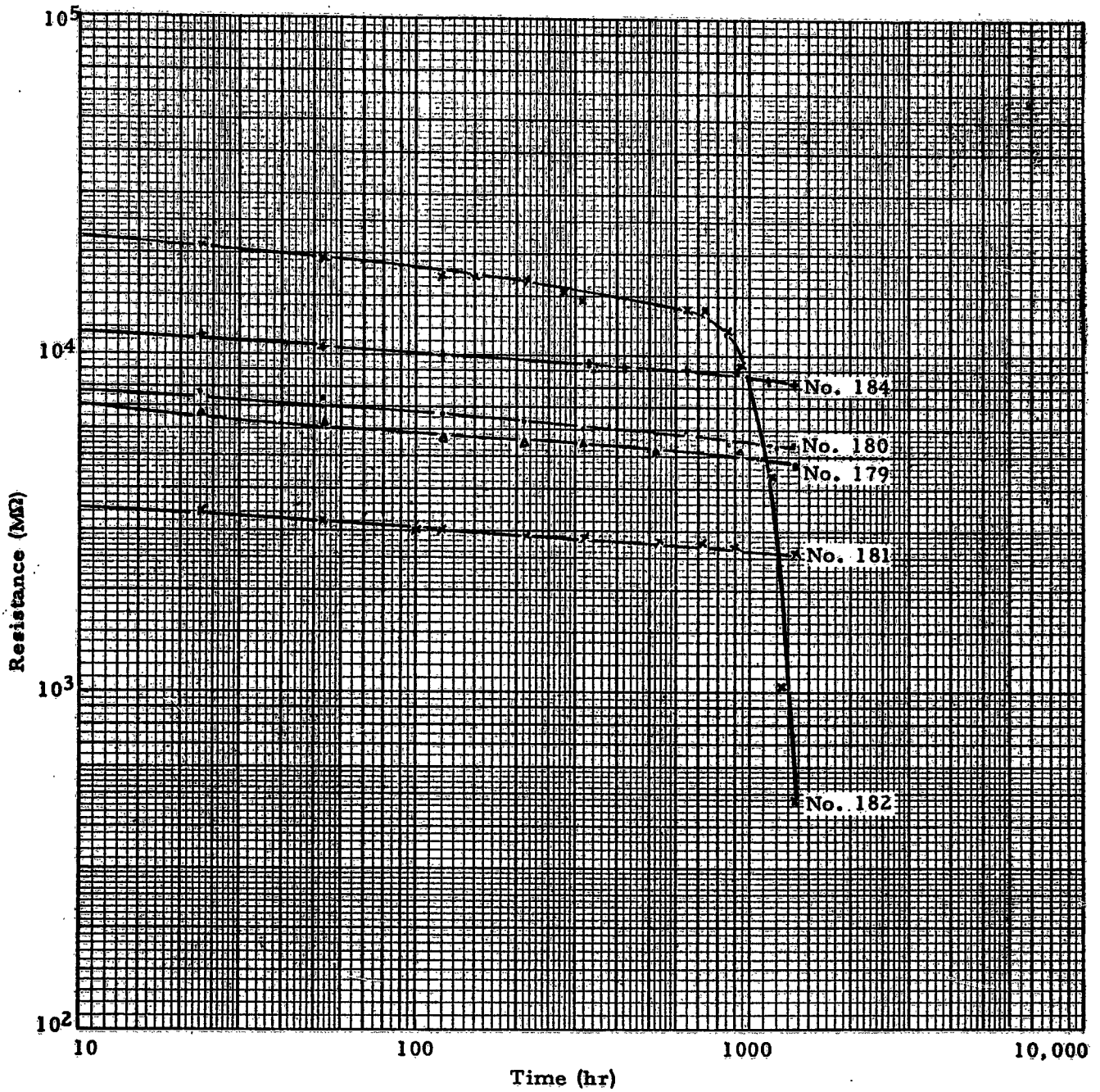
Figure 10



RESISTANCE VS TIME
FOR IMPROVED 0.01 μf C67 CASE SIZE I MONOLYTHIC CAPACITORS

Dielectric Thickness: 0.0025 in.
Conditions: 150°C, 220 VDC

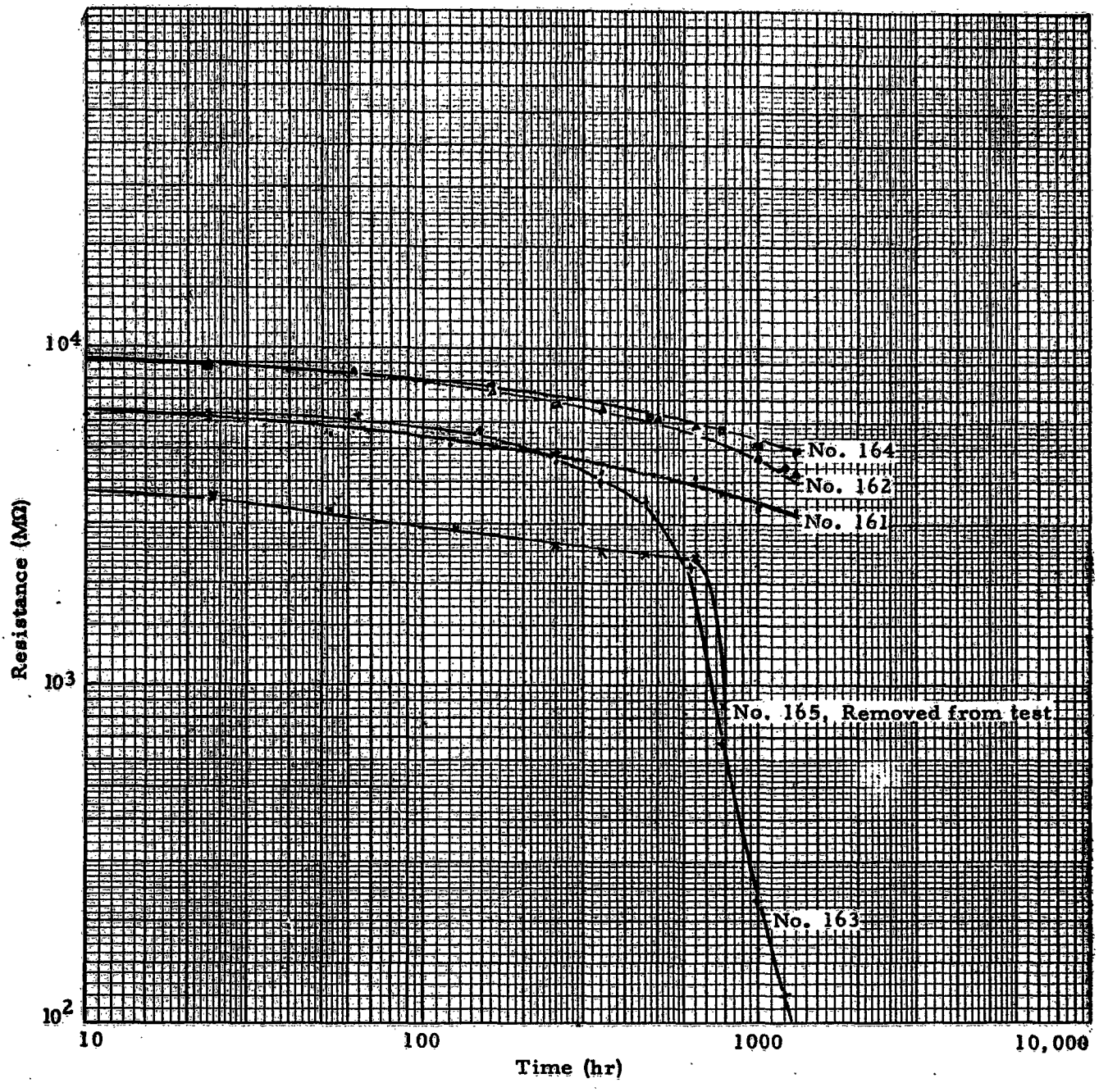
Figure 11



RESISTANCE VS TIME
FOR IMPROVED 0.01 μf C67 CASE SIZE I MONOLYTHIC CAPACITORS

Dielectric Thickness: 0.0025 in.
Conditions: 150°C, 400 VDC

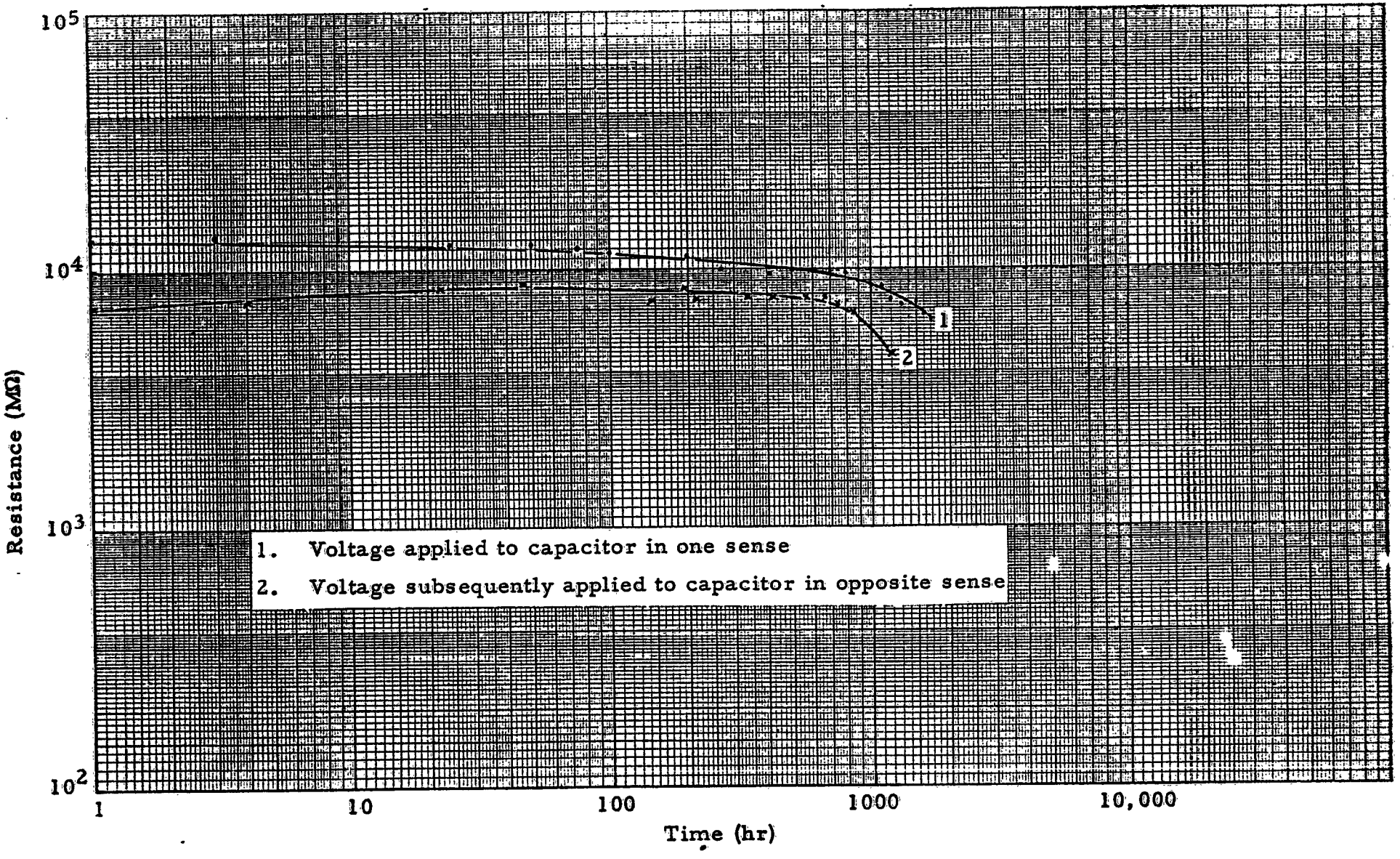
Figure 12



RESISTANCE VS TIME
FOR IMPROVED 0.01 μf C67 CASE SIZE I MONOLYTHIC CAPACITORS

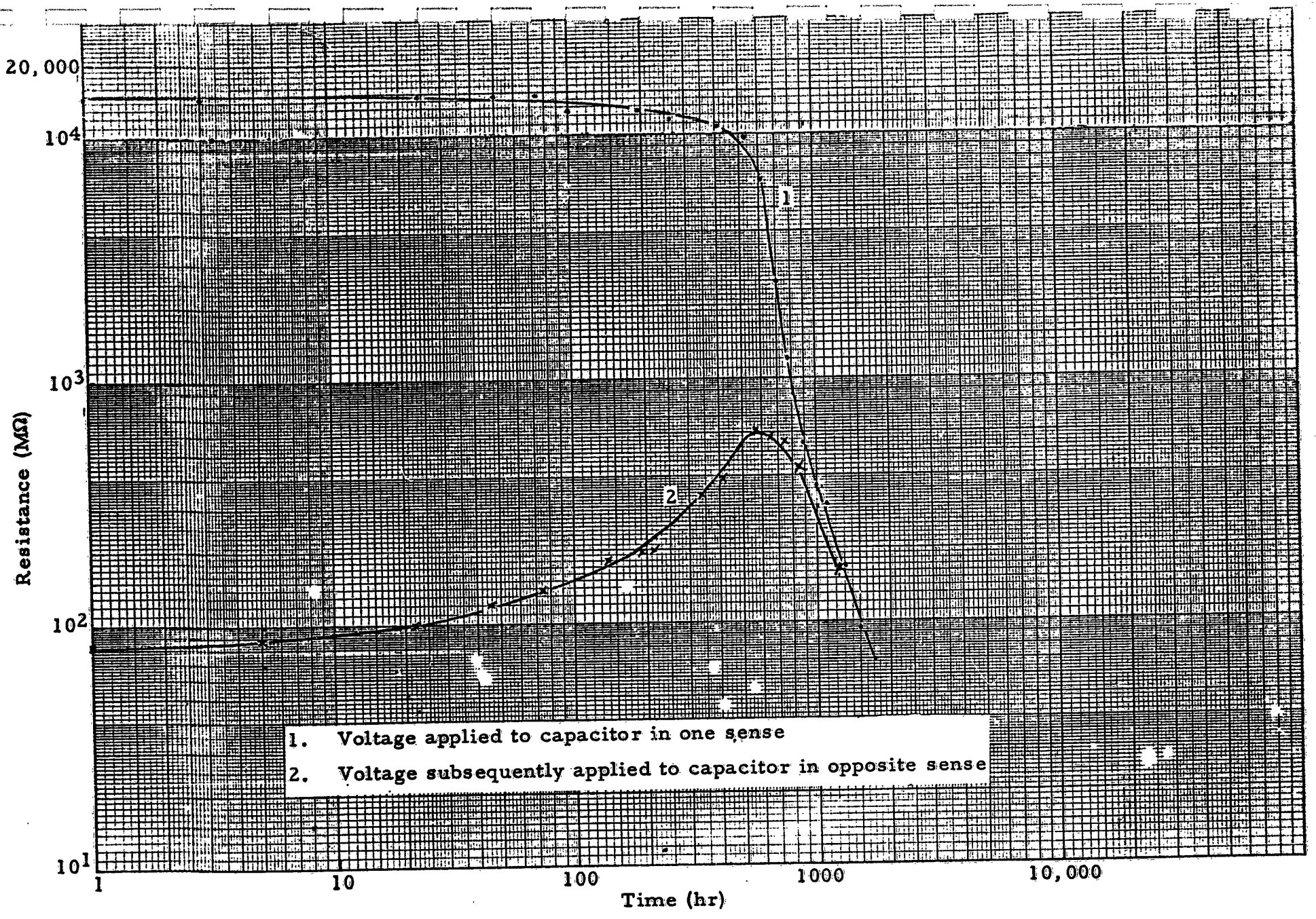
Dielectric Thickness: 0.0025 in.
Conditions: 150°C, 600 VDC

Figure 13



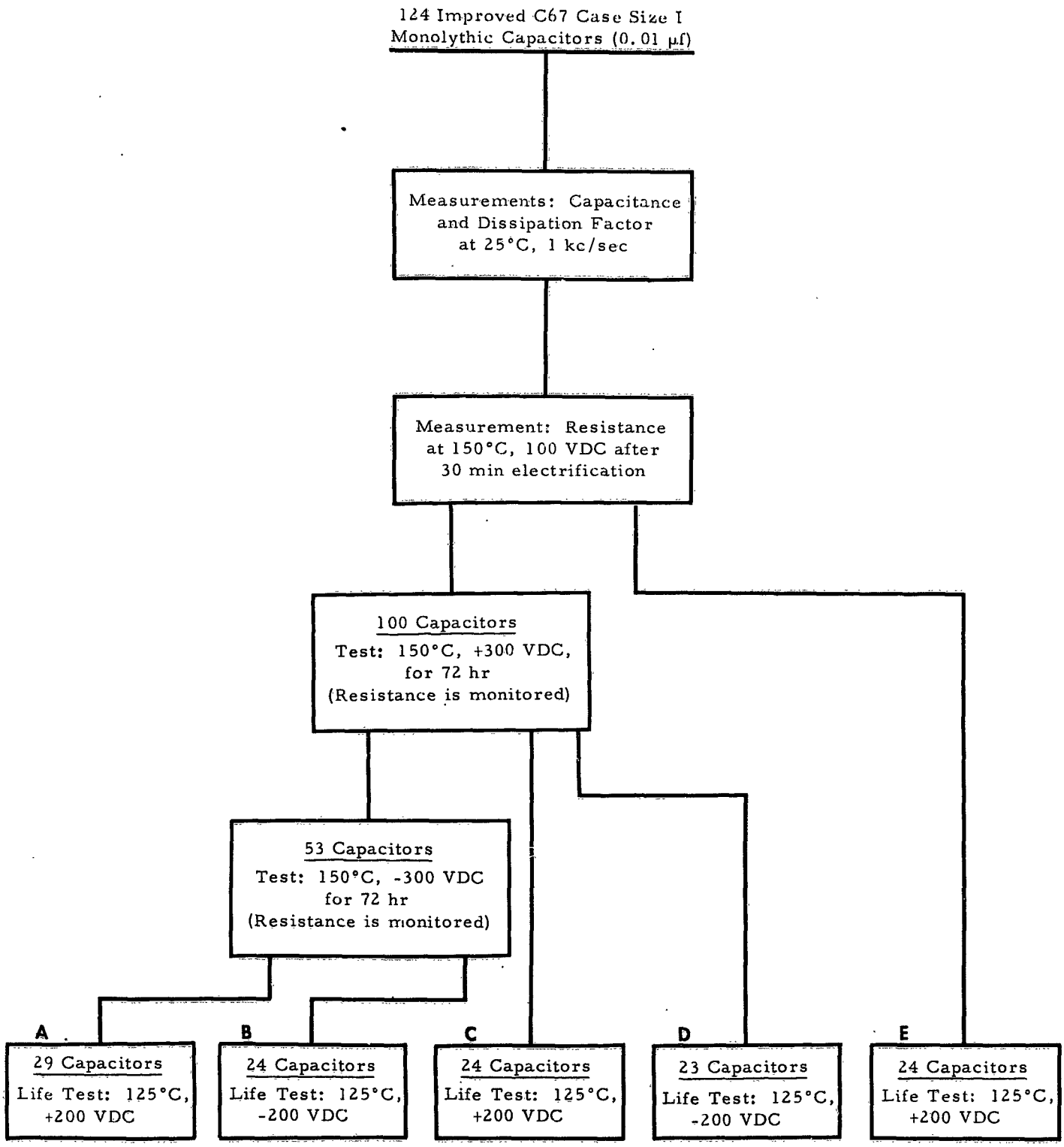
RESISTANCE VS TIME
FOR IMPROVED 0.01 μf C67 CASE SIZE I MONOLYTHIC CAPACITOR NO. 0164
Conditions: 150°C, 220 VDC

Figure 14



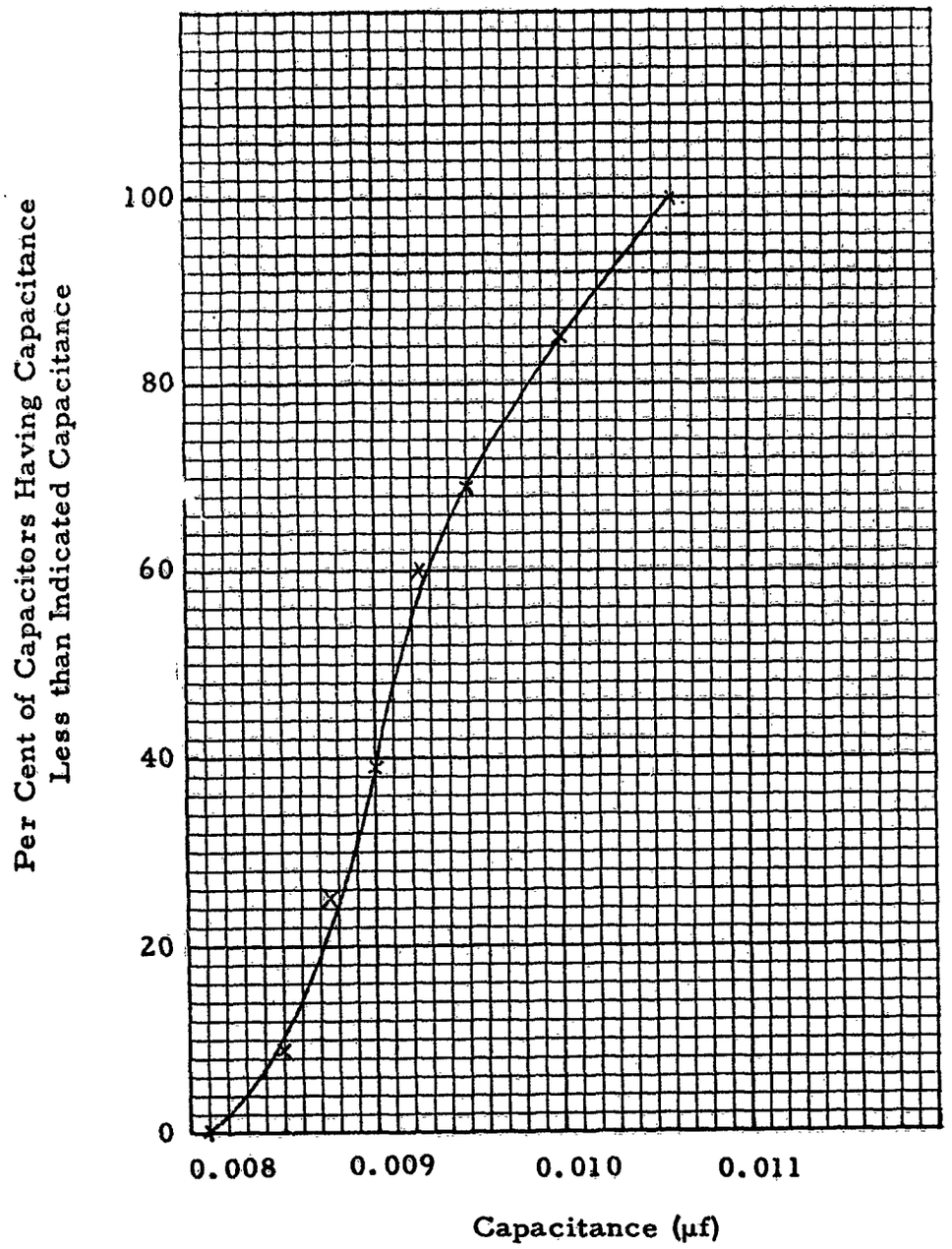
RESISTANCE VS TIME
FOR IMPROVED 0.01 μf C67 CASE SIZE I MONOLYTHIC CAPACITOR NO. 0169
Conditions: 150°C, 220 VDC

Figure 15



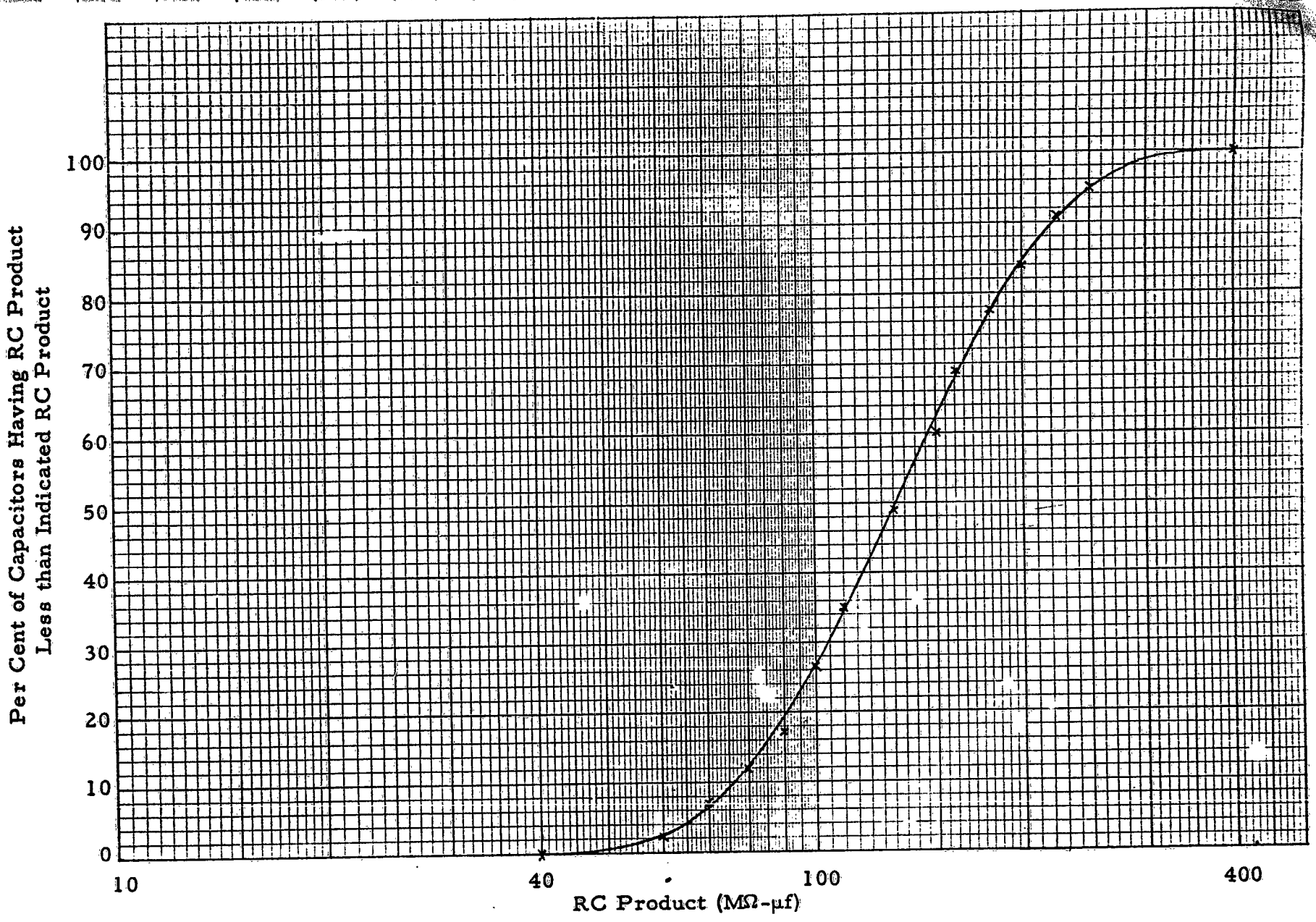
EXPERIMENT TO SELECT CAPACITORS HAVING POTENTIALLY LONG LIVES

Figure 16



DISTRIBUTION OF CAPACITANCES
FOR NEW, IMPROVED 0.01 μf C67 CASE SIZE I MONOLYTHIC CAPACITORS
Capacitance at 25°C, 1 kc/sec

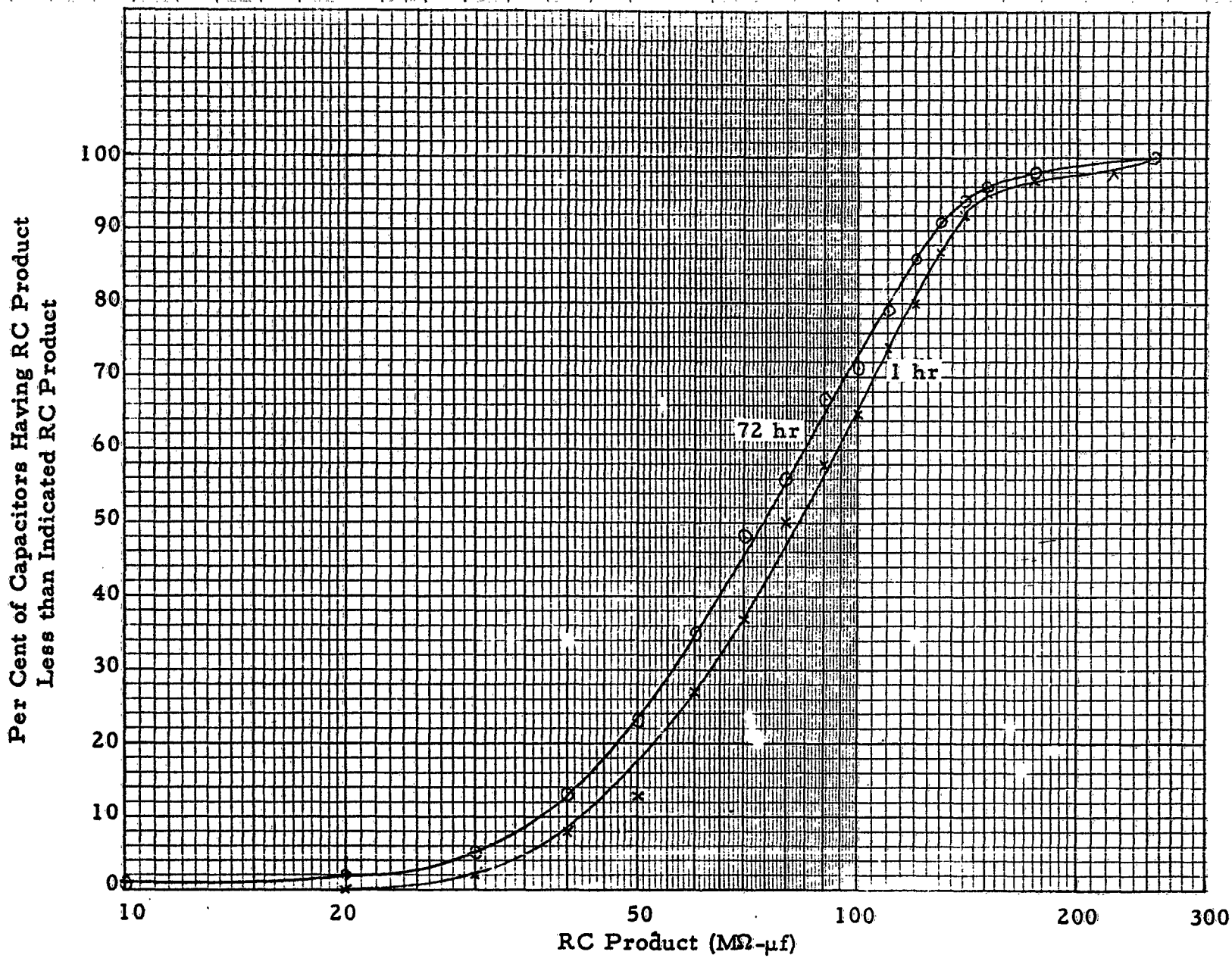
Figure 17



DISTRIBUTION OF RC PRODUCTS
FOR NEW, IMPROVED 0.01 μf C67 CASE SIZE I MONOLYTHIC CAPACITORS

R at 100 VDC, 150°C, 30 min electrification
C at 0.5 V_{rms}, 25°C, 1 kc

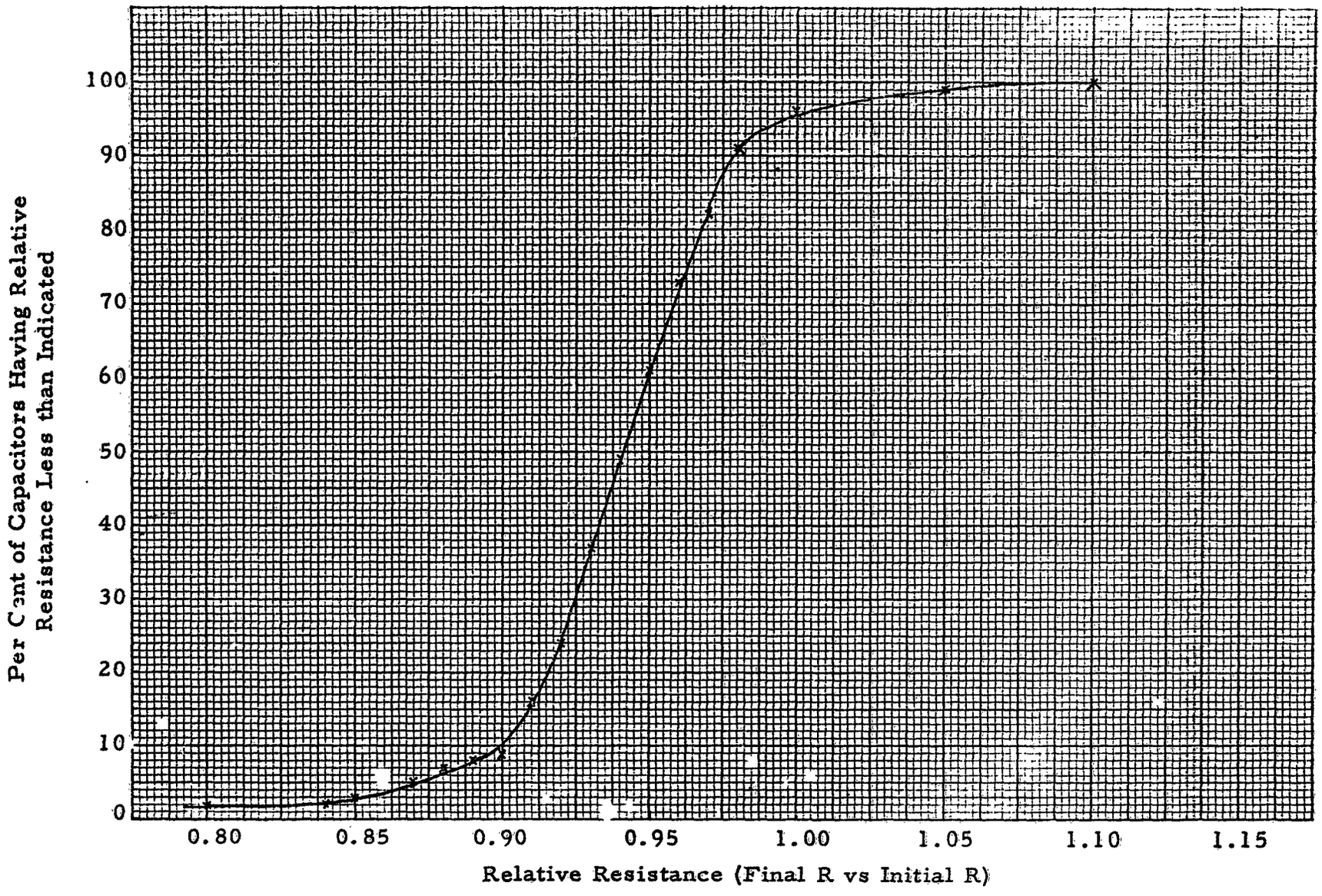
Figure 18



DISTRIBUTION OF RC PRODUCTS DURING INITIAL BURN-IN AT 300 VDC, 150°C
FOR IMPROVED 0.01 μf C67 CASE SIZE I MONOLYTHIC CAPACITORS

R at 300 VDC, 150°C
C at 0.5 V_{rms}, 25°C, 1 kc

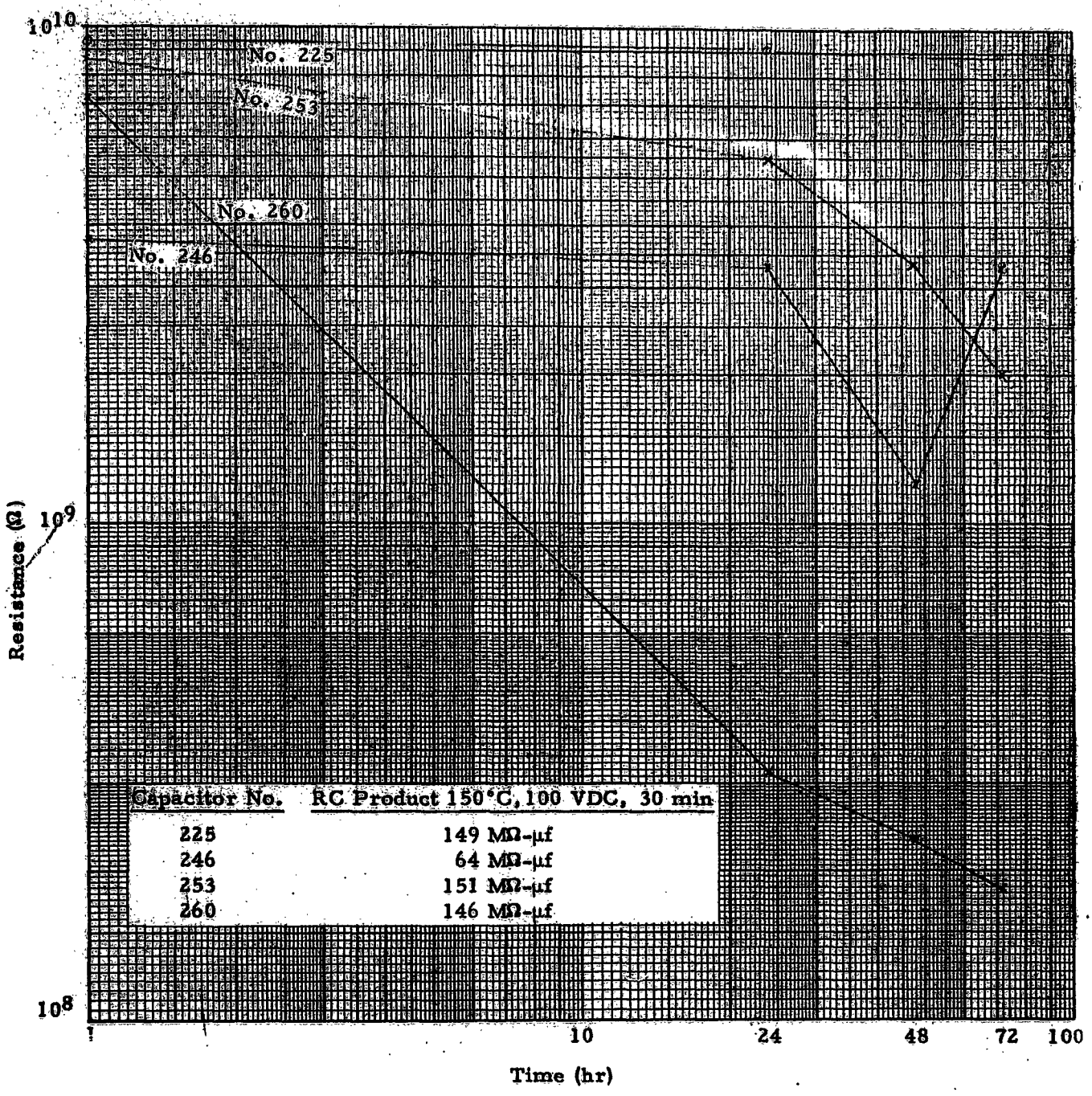
Figure 19



DISTRIBUTION OF RESISTANCE CHANGES DURING INITIAL BURN-IN FOR IMPROVED 0.01 μf C67 CASE SIZE I MONOLYTHIC CAPACITORS

(Resistance at 72 hr burn-in relative to resistance after 1 hr burn-in)
(Burn-in Conditions: 150°C, 300 VDC, 72 hr)

Figure 20

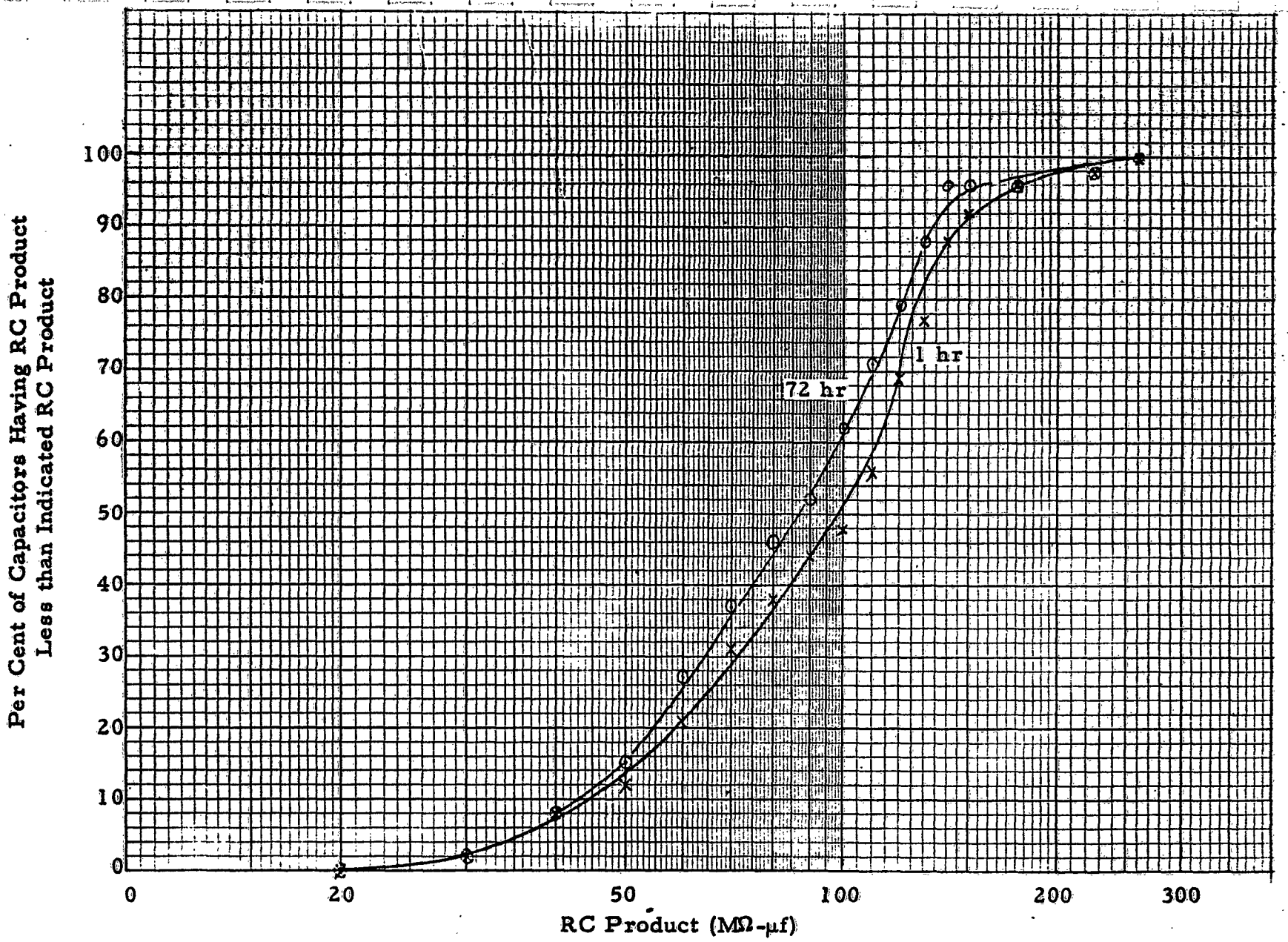


Capacitor No.	RC Product 150°C, 100 VDC, 30 min
225	149 MΩ-μf
246	64 MΩ-μf
253	151 MΩ-μf
260	146 MΩ-μf

RESISTANCE VS TIME DURING INITIAL BURN-IN
FOR IMPROVED 0.01 μf C67 CASE SIZE I MONOLYTHIC CAPACITORS

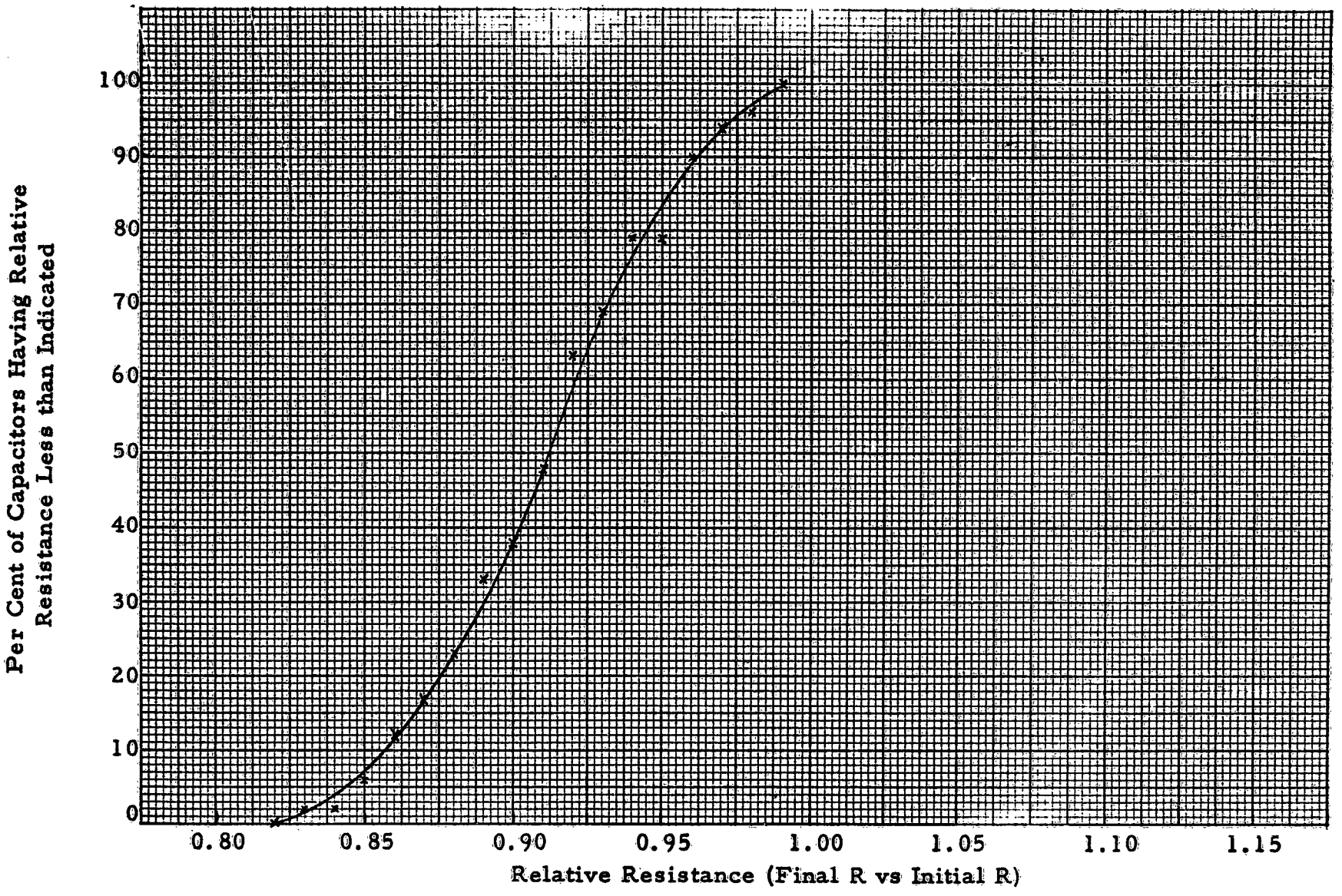
(Burn-In at 300 VDC, 150°C for 72 hr)

Figure 21



DISTRIBUTION OF RC PRODUCTS DURING "REVERSE" BURN-IN
FOR IMPROVED 0.01 μf C67 CASE SIZE I MONOLYTHIC CAPACITORS
R at 300 VDC, 150°C
C at 0.5 V_{rms}, 25°C, 1 kc

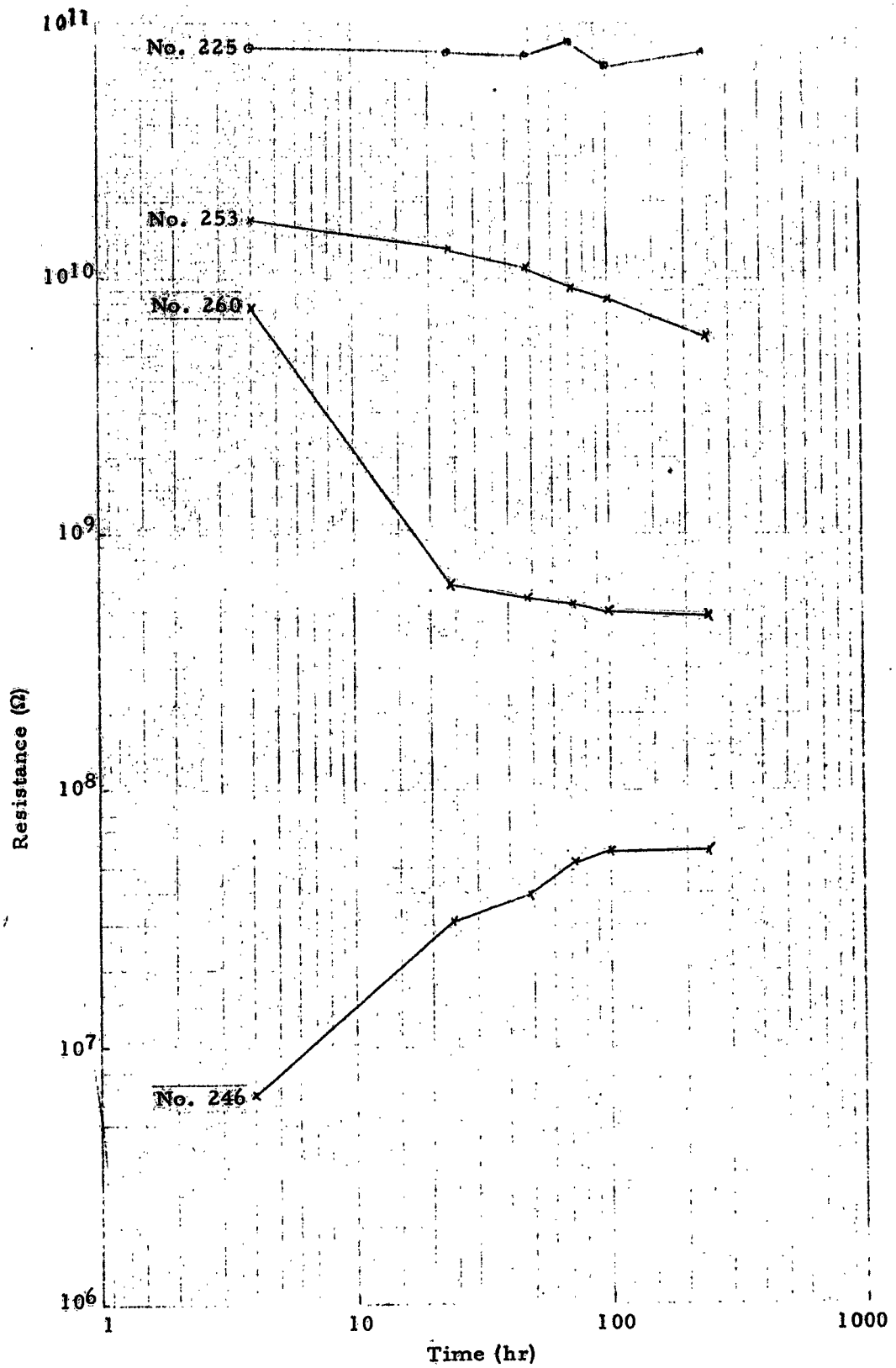
Figure 22



DISTRIBUTION OF RESISTANCE CHANGES DURING REVERSE BURN-IN FOR IMPROVED 0.01 μf C67 CASE SIZE I MONOLYTHIC CAPACITORS

(Resistance at 72 hr burn-in relative to resistance after 1 hr burn-in)
(Burn-in Conditions: 150°C, 300 VDC, 72 hr)

Figure 23



RESISTANCE VS TIME
FOR IMPROVED 0.01 μf C67 CASE SIZE I MONOLYTHIC CAPACITORS
Conditions: 200 VDC, 125°C

Figure 24

SECTION 5

CONCLUSIONS

(1) A slight change in the C67 composition has resulted in improved life test stability of the Case Size I Monolythic capacitors. This change has not affected the dielectric constant or temperature coefficient of dielectric constant.

(2) Characterizations on the improved capacitor with respect to leakage current and voltage, temperature and time were performed during this period. The steady-state electrical conductivity of both new and aged capacitors is non-ohmic. In general, conductivity is proportional to $V^{1.4}$. The thermal activation energy for electrical conduction for both new and aged capacitors is 1.05 eV. It appears that the life time of a capacitor subjected to direct voltage testing can be extended if, after a period of time, the voltage sense is reversed.

(3) An experiment has been started to examine the value of a selection technique for the detection of potential early failures from among the improved capacitors. The design of the experiment was greatly influenced by the characteristics of the obsoleted version of the capacitor and the life time formula developed for it. Results from the early stages of life testing indicate that capacitors which demonstrated resistance instability during screening will probably be the first to fail life test.

(4) The results of step-stress life testing of 27 capacitors of the obsoleted version indicate that time-to-peak-resistance at accelerated conditions is a meaningful indication of relative life times at less severe conditions of temperature.

(5) The life test of 753 C67 Case Size I Monolythic capacitors of the obsoleted version was continued. It would appear that a necessary but not totally sufficient condition for the prevention of early failure is that the resistance value at 150°C exceed 200,000 MΩ in the case

of 0.008 μf capacitors. Very low values of resistance, relative to the population as a whole, generally indicate low values of time-to-peak-resistance, which, according to the life time formula developed during the seventh quarter, is directly proportional to life time.

SECTION 6

PROGRAM FOR NEXT QUARTER

(1) Examination of the screening program for the selection of long-lived capacitors of the improved version will continue. Application of the approximate life time formula to the improved capacitor will be examined.

(2) The manufacture of 1500 0.01 μf capacitors of the improved version for voltage-temperature matrix testing will begin.

(3) The step-stress life testing of 753 capacitors will be continued.

SECTION 7

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1	Director U. S. Army Electronics Laboratories Fort Monmouth, New Jersey Attn: AMSEL-RD-P (Department Director)
1	Director U. S. Army Electronics Laboratories Fort Monmouth, New Jersey Attn: AMSEL-RD-PE (Dr. E. Both)
(Balance of copies)	Director U. S. Army Electronics Laboratories Fort Monmouth, New Jersey Attn: AMSEL-RD-PEE (J. Allen)

SECTION 8

IDENTIFICATION OF PERSONNEL

Personnel	Hours
W. Brierly	175.0
L. Burdick	90.0
N. Eror	80.0
C. LaCasse	42.0
W. Pfister	7.0
T. Prokopowicz	80.0
R. Rotolo	49.0
F. Schoenfeld	25.0
G. Shirn	3.0
D. Smyth	9.3
W. Tatem	156.0
H. Tourjee	10.5
Total	726.8

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Eighth Quarterly Report on
INTRINSIC RELIABILITY,
SUBMINIATURE CERAMIC CAPACITORS

Report No. 8, 30 June 1964 24 illustrations 3 tables
(Contract No. DA-36-039-SC-90705) DA Project No. 3A99-15-001

A slight change in the ceramic composition of the C67 Case Size I MONOLYTHIC® capacitor has produced improved current stability which promises longer life. Characteristics of the improved capacitor with respect to leakage and voltage, temperature and time, are presented. An experiment has been started to examine the efficacy of a selection technique for the detection of potential early failures among the improved capacitors. Life testing of 753 capacitors of the original version was continued.

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2. Contract No. DA-36-039-SC-90705

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