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Progress is reported on the investigation of high (113°F) and low (-89°F) temperature storage effects on standard RM cell structures. Basic physical chemistry of KOH-ZnO H_2O electrolyte system, physical constants, and specific conductance over a range of temperatures were studied. The equilibrium value for saturation of ZnO in KOH solution at room temperature and optimum and usable ranges of electrolyte composition for - 55°C operation were determined. Data are given showing the effects of ZnO content of the electrolyte on gassing characteristics of amalgamated zinc anodes. Work was continued with modified Haring cells for measurement of polarization characteristics of electrodes in the RM electrochemical system, and data resulting from the investigations were discussed and shown in graphical form.

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QUARTERLY PROGRESS REPORT ON LOW TEMPERATURE DRY CELLS

REPORT NO. 2

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October 1, 1948 to
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ABSTRACT

Experimental work, as described in the first quarterly report on this contract, to determine the effects of high (113°F) and low (-20°F) temperature storage effects upon standard RM cell structures was continued.

A study to determine the basic physical chemistry of the KOH-ZnO H_2O electrolyte system was continued. Physical constants and specific conductance were studied over a range of temperatures and compositions. The equilibrium values for saturation of ZnO in KOH solutions at room temperature were determined and the values so obtained were compared to those in available literature. The optimum and usable ranges of electrolyte composition for low temperature (-55°C) operation (based upon these studies) were determined. Methods of chemical analysis of electrolyte components are presented. Initial data showing the effect of the ZnO content of the electrolyte upon gassing characteristics of amalgamated zinc anodes is presented.

Continued work with the modified Haring cell for measurement of polarization characteristics of the electrodes in the RM electrochemical system is presented. Further necessary design changes are discussed. Results to date indicate that this latest design may be acceptable for polarization measurements, but more work will be required to determine its validity.

Further tests involving substitution of powdered metals for graphite in the mercuric oxide depolarizer were discontinued. Results of preliminary exploratory tests of other possible depolarizing agents for an alkaline cell are presented.

Results of work conducted to determine the low temperature discharge characteristics of standard RM structures are given: low temperature performance with standard commercial electrolyte formulae is compared to the performance with the optimum electrolyte compositions for low temperature use developed during the last quarter.

PUBLICATIONS AND REPORTS

Meteorological Battery (Signal Corps Contract No. W-35-039-sc-32025)

Scott's Standard Methods of Chemical Analysis Vol. I pp. 1064-1065
Vol. II pp. 2255-2257

Solubilities of Inorganic and Metal Organic Compounds Vol. I pp. 1589-1591
By Seidell

Treatise on Inorganic and Theoretical Chemistry Vol. IV pp. 521-535
By Mellor

Zeit. Anorg. Chemie Vol. 74 pp. 157-169 (1912)

Zeit. Electro-Chem. Vol. 33 pp. 134-144 (1927)

I INTRODUCTORY

The basic problem and contractual requirements for this investigation to improve the low temperature characteristics of the RM electro-chemical system were presented in the first quarterly report. In order to determine the low temperature possibilities and limitations of the RM electro-chemical system, it was necessary first to determine the factors that affect low temperature performance. A study of these factors was resolved into certain basic lines of approach. These were:

1. a study of the basic physical chemistry of the electrolyte system involving determination of physical constants, conductance measurements, and determination of electrode polarization characteristics

2. a study of possible means of improvement of the anode and cathode (depolarizer)

3. experimental work to determine the effects of high and low temperature storage characteristics of present cell structures

4. experimental work to determine low temperature discharge characteristics of present cell structures

5. development of new or improved structures suited for low temperature operation.

II TEXT

Investigation of the best high and low temperature performance of the RM electro-chemical system.

A. Experimental work to determine the effects of high and low temperature storage upon the standard RM cell structure.

1. Tests were initiated during the first quarter to determine the effects of low temperature, about -80°F (-60°C), storage upon standard RM cell structures of both pressed powder and foil anode type. Since the end of the first three month storage period was quite close to the time at which this report was written, results of these 3 months storage tests will be presented in the following quarter's report.

2. Similar tests were initiated during this quarter involving 113°F storage of standard RM cell structures of both pressed powder anode and foil anode type.

B. Electrolyte Study - Basic Physical Chemistry of the Electrolyte System.

1. Determination of the physical constants of the KOH-ZnO-H_2O system.

The thermal constitution diagram for the system KOH-H_2O (with no ZnO present) was established during the first quarter. During this quarter the effect of addition of ZnO to the KOH-H_2O system was determined by means of a similar series of measurements employing solutions containing fixed increasing increments of zinc oxide, namely 2%, 4%, 6%, and 8% by weight. The resulting thermal constitution diagrams are given respectively in Figures I, II, III, and IV appended.

The eutectic composition in the absence of zinc oxide is 31.3% (by weight) of KOH. When ZnO is added, the eutectic composition in the range studied is 30% (by weight) of KOH. All percentages of KOH in this investigation are expressed on an anhydrous basis. At -55°C, the lowest temperature range specified for cell operation in this study, the ranges of composition over which the electrolyte remains liquid (neglecting super cooling phenomena) are as follows:

0%	ZnO	----	28.5-35%	KOH
2	"	----	28-31.5	"
4	"	----	28-32.5	"
6	"	----	27-32	"
8	"	----	25.5-32.5	"

At the extremes in ZnO content, namely 0% and 8%, the spread in permissable variation of KOH content is greatest. A range of 28-31.5% KOH remains liquid at -55°C throughout the compositions studied.

All solutions were prepared by dissolving the ZnO in very hot, concentrated solutions of KOH in the desired ratio and then diluting to the desired composition. During the preparation of the above series of solutions it was observed that there was a lower limit of KOH concentration. The limiting value increased with increasing ZnO content. For this reason, the constitution diagrams progressively became more restricted below the eutectic. A study was therefore instigated to determine experimentally the saturation values for ZnO in KOH at room temperature.

The saturation values for commercial ZnO (Reagent grade-Merck & Co.) in various compositions of KOH were determined in the following manner. One hundred gram samples of various KOH contents were prepared in small Erlenmeyer flasks and cooled to room temperature. A large excess of ZnO was added and the flasks were tightly stoppered. The solutions were agitated periodically and the resulting liquid phases were analyzed after a four hour or twenty-four hour, and after a one week period.

The analyses of the various solutions after one week were generally close duplicates of the earlier analyses indicating that an equilibrium had been established. These results, representing saturation values for commercial ZnO in KOH at room temperature (25°C), are given in Table I appended.

A survey of the literature indicated that considerable previous work had been done to establish the equilibrium or saturation values for zinc oxide and zinc hydroxide in potassium and sodium hydroxide solutions up to three molar concentrations of the alkali. The solubility of zinc hydroxide in potassium hydroxide is expressed in equation form by J. Moir (Ref. Treatise on Inorganic and Theoretical Chemistry by Mellor - Vol. IV pp. 528); additional data is presented by O. Klein (Ref. - ibid - p. 527, and (Ref. Zeit. Anorg. Chem. Vol. 74 pp. 157-169 (1912)). Their data is expressed upon a molar basis. Moir's equation is as follows:

$$y = \frac{.004X(79X+6)}{X+2}$$

X = moles/liter KOH y = moles/liter ZnO

These investigations stated that three distinct solubility curves for zinc hydroxide were obtained - a most soluble and least stable form (A), an intermediate form (B), and a stable and least soluble form (C).

Work by Muller and associates (Ref. Zeit. Electro-Chem. Vol. 33, pp. 134-144 (1927)), identified the forms A, B, and C respectively as $Zn(OH)_2$, $ZnO.\frac{1}{2}H_2O$ and ZnO.

A study was therefore conducted to determine the solubility of $Zn(OH)_2$ in KOH solutions. The first method was to introduce zinc into solution electro-chemically by anodic attack. A group of five solutions with graded KOH concentrations were placed in 50ml. beakers. Sheet zinc anodes and pressed cylinders of HgO-C cathodes were employed as electrodes. A depolarizing cathode was employed to preclude deposition of zinc upon the cathode. The five cells were connected in series with a six volt rectifier and were discharged under an impressed D.C. current of $\frac{3}{4}$ ampere for three consecutive eight hour runs until a copious white precipitate formed - presumably ZnO. Electrodes were replaced several times during the test. The solutions were then analyzed for KOH and ZnO. Results of this investigation are presented in Table II appended, which shows that the solubility of ZnO increases with increase in KOH concentration.

The second method involved precipitation of zinc hydroxide from a zinc chloride solution by addition of potassium hydroxide. The precipitate was filtered and washed thoroughly with distilled water: it was then added in excess to solutions of varying KOH concentrations and mixed periodically for 24 hours. The resulting solutions were then analyzed for KOH and ZnO. Results from these tests are given in Table III appended.

Values obtained during these latter investigations correspond closely to the values reported by O. Klein., for the B form solubility curve. Figures obtained experimentally for saturation values of commercial zinc oxide correspond closely to those reported by Klein for the stable form C. A comparison of values obtained experimentally and those previously reported by Klein is given in Figure VI appended. Noting that the lower limit of the liquid range obtained on solution make-up for freezing point determinations agreed well with Meir's equation, it was decided to use this equation to express the equilibrium of form A.

A summary solubility diagram for the KOH-ZnO-H_2O system including isothermal freezing point curves and experimentally determined room temperature curves for ZnO is presented in Figure VII appended. It can readily be seen from this figure that the liquid range for operation at -55°C is quite restricted being bounded by the isothermal freezing curves and the stable (C) form solubility curve at room temperature. Based upon this data, the optimum KOH concentrations ranges from 0 to 4.5% at the lower and 0 to 5.9% at the higher KOH concentration.

2. Conductance measurements upon the electrolyte system $KOH-ZnO-H_2O$.

Variation of conductance with temperature and with composition in the system $KOH-H_2O$ (with no ZnO present) was established during the first quarter. A conductivity cell with a cell constant of 0.93 was employed for these measurements. During this quarter the effect of addition of ZnO to the $KOH-H_2O$ system was determined by means of a similar series of measurements employing solutions containing fixed increasing increments of zinc oxide, namely 2%, 4%, and 8% by weight. Graphs of conductance values vs. composition and vs. temperature are given in Figures VIII, IX, X, and XI appended.

A recheck of the conductivity data from these various solutions was made at temperatures of -30°C or lower, employing a conductivity cell with a cell constant of 0.1175. These more accurate values for the specific conductivity of electrolyte compositions containing 0, 2, 4, and 8% by weight zinc oxide are given respectively in Figures XII, XIII, XIV, and XV. A summation of results of conductance measurements of usable electrolyte compositions at temperatures of -40°C, -50°C, and -55°C is presented respectively in Figures XVI, XVII, and XVIII appended.

To illustrate the marked effect of variation of electrolyte composition and temperature upon conductivity the following conclusions or generalizations were drawn from the conductivity measurement data:

a. The following rates of increase of conductivity with temperature increase were calculated as average values over the range of electrolyte compositions studied.

Range°C	Average % Increase/1°C Temperature Change
+10°to+20°	2.7%/1°C
-20°to-10°	4.7%/1°C
-30°to-40°	9.6%/1°C

b. The optimum concentration, that with maximum conductivity, varies with the temperature range.

Range°C	Maximum Conductivity Value
-20°to 0°	35% KOH
0°to-20°	22% KOH
-20°to-50°	as near liquidus line on the low side of the eutectic composition as possible (Ref. Figures VIII, IX, X, and XI appended), conductivity in the usable (liquid) range is best at this liquidus line, but sufficient variation of composition may occur in electrolyte manufacture to produce a solution which freezes if one employs a composition corresponding to values on this liquidus line. Therefore, higher KOH concentrations as near to this value as are allowed by

practical considerations are considered optimum values upon this basis of selection.

c. The conductivity decreases with increasing zinc oxide content. For example, at -50°C, an addition of 2% by weight ZnO to a $KOH-H_2O$, solution decreases conductivity by 24% of the initial value. An addition of 4% ZnO decreases the conductivity by 40% and an addition of 8% ZnO decreases the initial value by 60%.

d. From the standpoint of conductivity, the optimum electrolyte composition for operation at -55°C is 29-30% KOH with the minimum ZnO content compatable with good storage characteristics.

In the work on effect of addition agents upon electrolyte conductivity in the RM system conducted last year (Ref. P. R. Mallory & Co., investigations titled Meterorological Battery), it was concluded that none of the large number of addition agents tested significantly improved cell conductivity. The most promising one tested at that time was potassium iodide. A recheck of the effects of KI addition was made upon an electrolyte containing 29.9% by weight of KOH and 0% ZnO. Results of this check are given in Table IV appended. The addition of potassium iodide generally tended to decrease conductance.

3. Chemical Analysis of Electrolyte (Methods)

Analyses for potassium hydroxide and potassium carbonate in a syster with no zinc oxide present were conducted by standard methods (Ref. Scott's Standard Methods of Chemical Analysis Vol. II pp. 2255-2257). With zinc oxide present in the electrolyte the potassium hydroxide content was determined by direct titration to the phenolphthalein endpoint with standard hydrochloric acid. Potassium carbonate content could not be determined by the direct titration method when ZnO was present so this value was estimated from the K_2CO_3 content determined by direct titration on the KOH source employed in solution makeup. Zinc oxide content was determined by means of a ferrocyanide titration procedure. (Ref. - ibid. Vol. I pp. 1064-1065.)

Results obtained by means of the above methods of analysis were compared to those obtained by direct electrometric titration of the same $KOH-ZnO-H_2O$ solution. A Beckman Model N, p H Meter (National Technical Laboratories, South Pasadena California) was employed in this electrometric titration. The titration curve so obtained is presented in Figure XIX appended. As can be seen from the following figures the data was generally in good agreement.

	Colormetric Endpoint Chemical Analyses	Direct Electro-metric Titration
%KOH	48.1	48.1
%ZnO	7.94	7.86*

*Note: This value was calculated from the endpoint pH(3.0) where the last visible traces of ZnO redissolved in the hydrochloric acid employed in titrating.

4. Effect of Zinc Oxide Content of the Electrolyte upon the Gassing Characteristics of Amalgamated Zinc Anodes

Since the work upon conductivity of electrolyte indicated that the optimum electrolyte composition for cell operation at -55°C would contain the minimum ZnO content commensurate with good storage characteristics, a test was initiated to determine the effects of ZnO content of the electrolyte upon gassing characteristics of amalgamated zinc anodes. In these tests, zinc samples were immersed in electrolyte and the gas was collected in calibrated centrifuge tubes. A 55°C storage temperature was used for these investigations. The effects of 0%, 1%, and 6% ZnO in a solution of 30% KOH (eutectic) were compared over a four week period. Sheet zinc about .015 inches thick with 2 square inches of surface area was used: amalgamation was 10% by weight and was effected by a solution containing 4% $Hg(CN)_2$, 10% KOH. Tests were conducted in triplicate.

Results of these tests are given in Figure XX appended. These data clearly show that some ZnO was necessary to suppress gassing but that no greatly significant difference between 1% and 6% ZnO was evident in the four week period. A further set of similar tests is now in progress comparing 0.5%, 1%, 2%, and 6% ZnO contents.

5. Polarization measurements in the RM Electro-Chemical System.

A proposed modified Haring cell design for measurement of electrode polarization in the RM electro-chemical system was presented in our first quarterly report. The cell design, which employed removable reference electrodes that are inserted into the electrode system only during the time of actual measurement, was judged to offer possibilities for such measurements. Further work was conducted during this quarter to determine the validity of this structure.

Initial work was conducted at room temperature using standard electrolyte consisting of 75 grams KOH, 10 grams ZnO, and 100 grams water. After a few tests it became evident that improved end electrode contact was mandatory if consistent, useful results were to be obtained. Therefore the cell end plates were redesigned to offer a cavity into which the powder electrode component could be consolidated so giving a more substantial duplication of commercial cell structure. This redesign of cell end plates is shown in Figure XXII appended.

Tests were conducted at room temperature using pressed powder electrode components in this redesigned structure. Reasonable but not exact duplication of results was obtained during these tests. Current densities of 100 and 150 milliamperes per square inch were employed (calculated on apparent or cross sectional surface area). Cathode polarization data from these tests is given in Figure XXIII appended; anode polarization data from these tests is given in Figure XXIV appended. These results show that under these conditions of electrolyte composition, current density, and temperature, cathode polarization is more significant than anode polarization, both initially and throughout a 24 hour discharge period.

Room temperature variation within the range 23-29°C was found substantially to influence measurements. An increase in temperature decreased polarization values and solution IR drop (VG-G) by a noticeable amount. Therefore for accurate measurement a controlled temperature bath is necessary. Neglecting effects of temperature variation, a steady increase in IR drop (VG-G) was noted during these tests. This effect could be ascribed to the increase in ZnO content of the electrolyte content resulting from anode solution during the discharge period. Increase in ZnO content during three runs at a 20 milliampere drain rate on this cell for 21 to 24 hour discharge periods are tabulated below:

Initial Composition 75-10-100	Composition At End of 21 Hour Run	Composition At End of 24 Hour Run	Composition At End of 24 Hour Run
%KOH 34.35%	33.65%	33.0 %	33.2 %
%ZnO 5.4 %	8.05%	8.14%	8.82%

These changes in solution composition very likely affected the polarization data as well as the solution conductivity and would have even greater significance if duplicated during low temperature operation.

A series of similar tests was started at 0°C using a current density of 100 milliamperes square inch and the same electrolyte. A pressed powder anode component still gave reasonable duplication of results but attempts to employ a sheet (disc) anode component consolidated into the well in the anode end plate did not give satisfactory duplication of results. The anode contact using sheet zinc was too erratic, and a further design change to accommodate this type of electrode was necessary. It was also noted that cathode polarization results were more variable at this temperature. This may have been due to the considerable variation in grain size of the production 5% graphite (regranulated) depolariser mix employed in the tests so far.

A further redesign of the Haring cell structure was therefore made. In this new design either pressed powder or sheet (disc) electrode components can be used alternately as desired at either end of the cell without altering the internal dimensions of the structure. The cell diameter was increased to 0.75 in. from the original 0.50 in. value and the internal lucite sleeves employed in the earlier design were discarded.

This eliminated a possible source of error which was inherent in the original design intended for use with fixed (screen grid) reference electrodes and which was not necessary or desirable with the present system of removable reference electrodes. This latest design of the polarization cell is shown in Figure XXV appended.

GENERAL CELL DIMENSIONS

Distance between reference electrodes and reference electrode to cell electrode	0.75 in.
Overall distance between anode and depolarizer	2.25 in.
Cell diameter	0.75 in.
Cross sectional area of cell	0.442 sq. in.
Volume of electrolyte	about 20 ml.

A series of duplicating tests was conducted at 0°C using standard electrolyte (75 grams KOH, 10 grams ZnO, 100 ml water) and a current density of 45.3 milliamperes square inch (a 20 milliampere drain rate for this redesigned cell). Sheet disc zinc anodes previously amalgamated with dilute $Hg(CN)_2$ solution were employed in these tests. Depolarizer components were pressed from a 5% C regranulated mix which had been separated by means of standard sieves to a size passing a 60 mesh screen. Cooling was effected by means of an agitated dry ice-distilled water bath in a Dewar flask. (Note: The conductivity of this dry ice - water bath was of relatively insignificant magnitude compared to that of the electrolyte at the same temperature. After one week's use it was about 1/3000th of the conductivity of the electrolyte).

Results of cathode polarization measurements on four duplicating runs are presented in Table V appended. Results of anode polarization measurements are given in Table VI appended. Anode polarization values are generally in good agreement. Some variation in results on duplicating runs is experienced but mean and average values are generally quite close and the maximum variation from mean values is small. Cathode polarization values are more erratic and less in agreement than anode polarization values, but considering the possible variations which may occur in the pressed powder cathode the data is reasonably in good agreement. A graph of this polarization data from duplicating measurements at 0°C is given in Figure XXVI appended. As can be seen from this graph, cathode and anode polarization are generally of the same order of magnitude with this solution composition, current density, and temperature.

C. Investigation of Depolarizer Improvement

1. Variation of particle size of constituents.

Some tests to determine the effect of particle size of graphite and mercuric oxide upon rate of migration and cell operation

were planned and a test upon "Dag" ultra fine graphite (product of Acheson Colloids Corp., Port Huron, Michigan), was initiated. This subject is now being investigated under government contract, (Ref. P. R. Mallory & Co., Inc. investigations entitled General Improvement of the RM Primary Cell), and further work in the immediate future is planned under this contract with respect to effect upon cathode polarization.

2. Substitutes - powdered metals for graphite in HgO depolarizer.

Results of some preliminary tests involving powdered iron and nickel in place of graphite were reported during the first quarter. Since these modifications do not improve depolarizer conductivity, since relatively large amounts (compared to graphite) are necessary to obtain reasonable cell operation, and since mechanical strength, coherence of depolarizer, and chipping characteristics were poor, further work along these lines has been discontinued for the present.

3. Depolarizers other than mercuric oxide.

A list of inorganic compounds which might conceivably be employed as depolarizing agents in the alkaline cell systems was presented in the first quarterly report. Preliminary exploratory tests of a very limited scope were conducted upon a number of these proposed materials.

Mercuric iodide, cupric iodide, and bismuth tri iodide were tested as depolarizing agents in 1R structures, the first mixed with 8% graphite and the others with 20% graphite. Inorganic asbestos type barriers were employed. Efficiency upon discharge was generally poor and discharge voltages were low. There were indications of depolariser decomposition upon storage and iodine compounds (identified by qualitative chemical analysis) formed on anode top and cell top. These results of limited exploratory tests indicate that these iodides are not particularly suitable as depolarizers in the standard RM structure.

Cupric sulfide and arsenous sulfide were tested in 1R structures in mixes containing 20% graphite; lead sulfide and mercuric sulfide were tested in the same manner in mixes employing 8% graphite. Discharge characterisites, voltage and efficiency of utilization, were generally poor. There were tendencies for decomposition and gassing during storage and sulfide compounds (identified by qualitative chemical analysis) formed on the anode and cell top. These results indicate that these sulfides are not well suited for use as depolarizers in the standard RM structure.

Silver permanganate - mixed with 10% graphite - was tested as the depolarizer in 1R structures. Some cells were constructed without barriers; in others a very thin dip coating of paraffin was employed to coat the depolarizer as recommended by the Ruben Laboratory. Initial open circuit voltages of 1.9-2.0 were obtained but values decreased to 1.8 in 48 hours. All cells swelled badly in 24 hours; this swelling

accompanied marked expansion of the depolarizer component. Some solution of the depolarizer occurred resulting in a blue-green colored solution (probably due to MnO_4^{-2} Ion). Anodes were generally blackened and corroded. Performance during discharge was poor. These exploratory tests indicated that silver permanganate was not well suited for use in the standard RM structure.

Silver oxide, mixed with 8% graphite, was investigated as a depolarizer in the 1R structure. Use of dialysis paper barriers led to quick failure on discharge testing and decomposition of the oxide during room temperature storage. Removal of the barrier markedly improved the performance of the structure on discharge testing. Use of inorganic (asbestos type) barriers gave results on discharge which were generally comparable to results without barriers. Results of initial discharge tests on 1R structures made without barriers are given in Figure XXI appended. Efficiency of cathode utilization on cells containing a large excess of zinc anode are plotted against external load during discharge. (Results represent average values for six cells at each load.

Silver oxide is the only prospective depolarizer so far investigated which seems worthy of any further study. It, however, seems to possess several disadvantages besides relative cost when compared to mercuric oxide -- namely, apparent lower efficiency under the same discharge conditions (on a 1R cell discharged through 65 ohms -- 68% efficiency for Ag_2O vs. 90% for HgO), a lower pressed density under equivalent pressures, and comparative instability (requiring use of more stable barrier materials). Further investigation will be necessary to obtain a valid appraisal of its applicability to the alkaline cell system.

D. Structures

It has been anticipated that any cell design suitable for low temperature operation should possess the maximum possible electrode area per unit volume and the closest practical electrode spacing. The eventual use of one of the following structures is, therefore, implied: a concentrically wound structure, an interwoven stack structure, or a parallel stack structure. Prior to initiating work with these designs, some work was conducted during this quarter to compare the performance at low temperatures of standard RM cell structures containing standard electrolyte formulae and the electrolyte formulae chosen during this period upon the basis of conductivity and freezing point measurements as being optimum for low temperature usage.

1. Performance of standard RMB-4 structure at low temperatures.

Standard RMB-4 cells were continuously discharged through 30 ohms loads at -20°C, -40°C, and -55°C. These cells are specified as containing electrolyte in the KOH-ZnO-H_2O ratio 100-13.2-100. The discharge curves so obtained are presented in Figure XXVII appended. Even at a temperature as high as -20°C the initial voltage on load was

at or below the standard 0.9 v. cut off value.

2. Performance of 1R cell structures at low temperature.

Three different electrolyte formulae were compared at low temperatures in a similar manner discharging continuously through 65 ohms loads at -20°, -40°, and -55°C. The electrolyte compositions employed were (1) standard 75-10-100 (34.5% KOH, 5.4% ZnO), (2) 57-8.3-100 (29.7% KOH, 5% ZnO), (3) 54-1.6-100 (29.7% KOH, 1% ZnO). The two latter formulae represent a near eutectic KOH concentration with, respectively, the near maximum amount of ZnO soluble at room temperature as stable form C - (Ref. Figure VII) and a low concentration which may be sufficient to give acceptable storage characteristics. The discharge curves so obtained are given in Figure XXVIII appended. The curves for open circuit voltage vs. temperature using a General Electric voltmeter with an internal resistance of 15,000 ohms (5,000 ohms/volt) are given in Figure XXIX appended. These results clearly indicate that in this structure the eutectic KOH concentration performs better at low temperatures than the standard production electrolyte formula; they also show that low temperature performance improves somewhat with decreasing ZnO content. With this structure the performance at low temperature is very poor and the differences between various electrolyte formulae are comparatively small.

3. No. 4 Gel electrolyte button cell structure with perforated disc type anode.

These cells were constructed employing 2 anode discs of .020 in. thick zinc, amalgamated with $Hg(CN)_2$, KOH solution. The theoretical rated capacity based upon anode weight is approximately 3.2 ampere hours. Gel electrolytes were made with 5 grams of CMC (Carboxy-methyl cellulose) per 100cc of electrolyte and were vacuum impregnated into the structure to assure complete filling. Two electrolyte formulae were compared, (1) 75-10-100 and 54-0-100 (30% KOH, 0% ZnO). These cells were compared on discharge through a 30 ohms load at temperatures of -20°C, -40°C, and -55°C. The discharge curves so obtained are given in Figure XXX appended. Performance was generally poor but was distinctly better with the 54-0-100 than with the 75-10-100 composition. Percentages of anode utilization calculated to a 0.9 v. cut off value were as follows:

Temperature	Electrolyte 75-10-100	Electrolyte 54-0-100
-20°C	5.0%	6.4%
-40°C	0%	0.7%
-55°C	0%	0%

4. No. 4 Gel elctrolyte button cell with convoluted anode.

These cells were constructed with anodes made of 0.150 in. X 0.006 in. zinc. A corrugated strip with 1/16 in. deep corrugations was spirally wound inside a flat strip to form the anode. These anodes were amalgamated with dilute $Hg(CN)_2$, KOH solution. The theoretical rated capacity based upon anode weight is approximately 2.25 ampere hours. Gel electrolytes were made with 5 grams of CMC per 100 ea. of electrolyte. A 75-10-100 formula was compared to a 54-0-100 formula by discharge continuously through 30 ohms at temperatures of -20°Cm -40°C, -55°C. The discharge curves so obtained are given in Figure XXXI appended. Performance was poor but was generally better than with the perforated disc anode. Performance with the 54-0-100 electrolyte formula was distinctly better than with the 75-10-100 composition. Percentages of anode utilization calculated to a 0.9 v. cut off value were as follows:

Temperature	Electrolyte 75-10-100	Electrolyte 54-0-100
-20°C	9.4%	24.5%
-40°C	0%	6.15%
-55°C	0%	0%

5. RMA 5/8 in. diameter (cylindrical) cell structure - gel electrolyte.

Standard penlight cells employing 100-16-100 electrolyte and 5/100 C.M.C. were compared to cells employing 54-0-100 electrolyte and 2.5/100 C.M.C. (the minimum amount forming a reasonably rigid gel) by continuous discharge at -20°C, -40°C, and -55°C through a 30 ohms load. The discharge curves so obtained are given in Figure XXXII appended. Performance was poor in general. The less rigid 54-0-100 electrolyte out-performed the standard 100-16-100 composition. Percentages of anode utilization calculated to a 0.9 v. cut off value were as follows:

Temperature	Electrolyte 100-16-100 5/100 CMC	Electrolyte 54-0-100 2.5/100 CMC
-20°C	2.4%	13.9%
-40°C	0%	3.25%
-55°C	0%	0%

Results with these various standard RM cell structures clearly indicate that there is extremely little possibility that any variations of these structures could fulfill the contractual requirements.

III PLANS FOR FUTURE WORK

The tests to determine the effects of low temperature (-80°F) and high temperature (113°F) storage upon standard Mallory RMB-3 and RMB-4

cells will be continued as scheduled during the first quarter. Similar tests involving Mallory radial crimp structures will be conducted, if deemed worthwhile, when enough samples of these newer structures are available.

Since the usable electrolyte composition range for low temperature operation (-65°F) has been established during this last quarter, it is next planned to conduct a polarization study to observe the effects of temperature variation, variation of electrolyte content within the workable range and variation of operating current density at the respective electrodes. When this data is available it will be possible to select the optimum electrolyte composition in the RM system for low temperature (-65°F) operation. It will also be possible to determine the electrode current densities workable at low temperatures and the respective electrode limiting current densities. This information will have a very important bearing upon the eventual low temperature cell structure and design.

Evaluation of the effect of ZnO content of the electrolyte upon gassing characteristics of amalgamated zinc anodes will continue.

Studies may subsequently be conducted to discover addition agents to improve electrolyte conductivity and minimize electrode polarization effects without adversely affecting the basic physical chemistry of the electrolyte system.

Investigation of depolarizer improvement will be continued. Effect of variation in granule size and particle size of ingredients upon polarization characteristics will be investigated.

A study is now in progress to find new or improved ways to prepare and to handle the electrode materials in thin sections. Investigations of methods for bonding depolarizer in thin, flexible sections and investigations to determine the best methods for increasing the true and apparent areas of zinc anode materials will be conducted.

A satisfactory, inert (probably inorganic) barrier and a suitable electrolyte absorbent must be found.

These investigations will lend themselves to incorporation in all previously mentioned new cell structures and will be instrumental in determining the eventual structure and design for a low temperature cell.

fs

P. R. MALLORY & CO., INC.

By T. C. O'Nan
T. C. O'Nan, Section Leader
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APPROVED:

R. M. Strain, Supervisor
Battery Research

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R. M. Boos, Chief Electro-Chemist
Research Division

TABLE I

SATURATION (EQUILIBRIUM) VALUES FOR
COMMERCIAL ZnO IN POTASSIUM HYDROXIDE
AT ROOM TEMPERATURE (25°C)

Nominal Original KOH Conc.	Analyses After 4 Hrs.		Analyses After 24 Hrs.		Analyses After 7 Days		
	%KOH	%ZnO	%KOH	%ZnO	%KOH	%ZnO	Spec. Gr.
5%	5.1	0.23	-	-	5.2	0.22	1.042
10%	10.0	0.88	-	-	10.4	0.80	1.093
15%	-	-	15.3	1.61	15.7	1.57	1.160
20%	20.0	2.97	-	-	20.2	2.69	1.225
25%	25.2	3.91	-	-	25.3	3.86	1.296
30%	-	-	29.7	5.33	29.9	5.17	1.368
35%	-	-	34.3	6.76	34.2	6.66	1.441
40%	-	-	40.4	8.87	39.3	8.80	1.535
45%	-	-	44.3	11.73	44.0	11.42	1.666
50%	-	-	46.8	14.53	47.3	14.48	1.747
50%	-	-	47.4	14.87	47.6	14.30	1.762

TABLE II

SATURATION (EQUILIBRIUM) VALUES FOR
ZINC OXIDE IN POTASSIUM HYDROXIDE
(ZnO INTRODUCED BY ANODIC SOLUTION)

Nominal Original %KOH	Spec. Gravity	% KOH	% ZnO
20	1.123	8.25	0.95
30	1.177	10.1	1.24
35	1.239	13.2	2.65
40	1.251	14.4	2.57
50	1.387	25.7	7.23

TABLE III

SATURATION (EQUILIBRIUM) VALUES FOR
ZINC HYDROXIDE IN POTASSIUM HYDROXIDE
(PRECIPITATED AND WASHED $Zn(OH)_2$ USED IN THE TEST)

Nominal Original %KOH	Spec. Gravity	% KOH	% ZnO
10	1.084	6.7	0.45
20	1.173	13.9	1.87
30	1.356	26.5	7.18
40	1.561	35.8	12.57
50	1.734	41.8	16.74

TABLE IV

EFFECT OF ADDITION OF POTASSIUM IODIDE
UPON CONDUCTANCE OF KOH-H_2O ELECTROLYTE

	Conductivity In Reciprocal Ohms			
Temperature °C	0% KI	2% KI	4% KI	6% KI
-40°	.0651	.0610	.0670	.0435
-45°	.0425	.0405	.0420	.0400
-50°	.0300	.0250	.0268	.0250
-55°	.0200	--	.0180	.0160

TABLE V

CATHODE POLARIZATION IN MILLIVOLTS AT 0°C

Drain Rate..... 20 Milliamperes
Current Density..... 45.3 Ma. Sq. In.
Depolarizer..... 13.0 Grams 5% Mix
Consolidated at
25,000 P.S.I.

Hours Discharge	Polarization Values Duplicating Tests 1	2	3	4	Average Value	Average to Nearest 5 Millivolts	Mean Value	Maximum Deviation From Mean Value
0	21	10.2	21.4	19.5	18.0	20	15.8	±5.6
½	7.5	5.9	7.3	14.0	8.8	10	9.9	±4.0
1	7.9	7.4	2.9	10.6	7.0	5	6.4	±3.5
1½	8.9	6.3	0.4	15.8	7.8	10	8.1	±7.7
2	10.4	6.9	2.9	14.5	8.9	10	9.2	±6.3
2½	5.5	4.8	3.5	18.0	7.9	10	10.8	±7.3
3	6.0	---	4.4	14.5	8.3	10	9.4	±5.0
3½	---	8.4	---	----	---	--	---	---
4	11.4	7.9	6.6	18.0	9.5	10	9.3	±8.7
4½	13.5	8.6	4.4	19.5	11.5	10	11.9	±7.5
5	11.6	13.8	5.9	22.0	13.3	15	13.9	±8.0
5½	10.5	12.5	7.2	19.0	12.3	10	13.1	±5.9
6	6.3	13.9	9.4	21.5	12.7	15	13.5	±7.2
6½	10.0	13.0	5.4	20.8	12.3	10	13.1	±7.7
7	8.0	----	6.2	----	----	--	----	---

TABLE VI

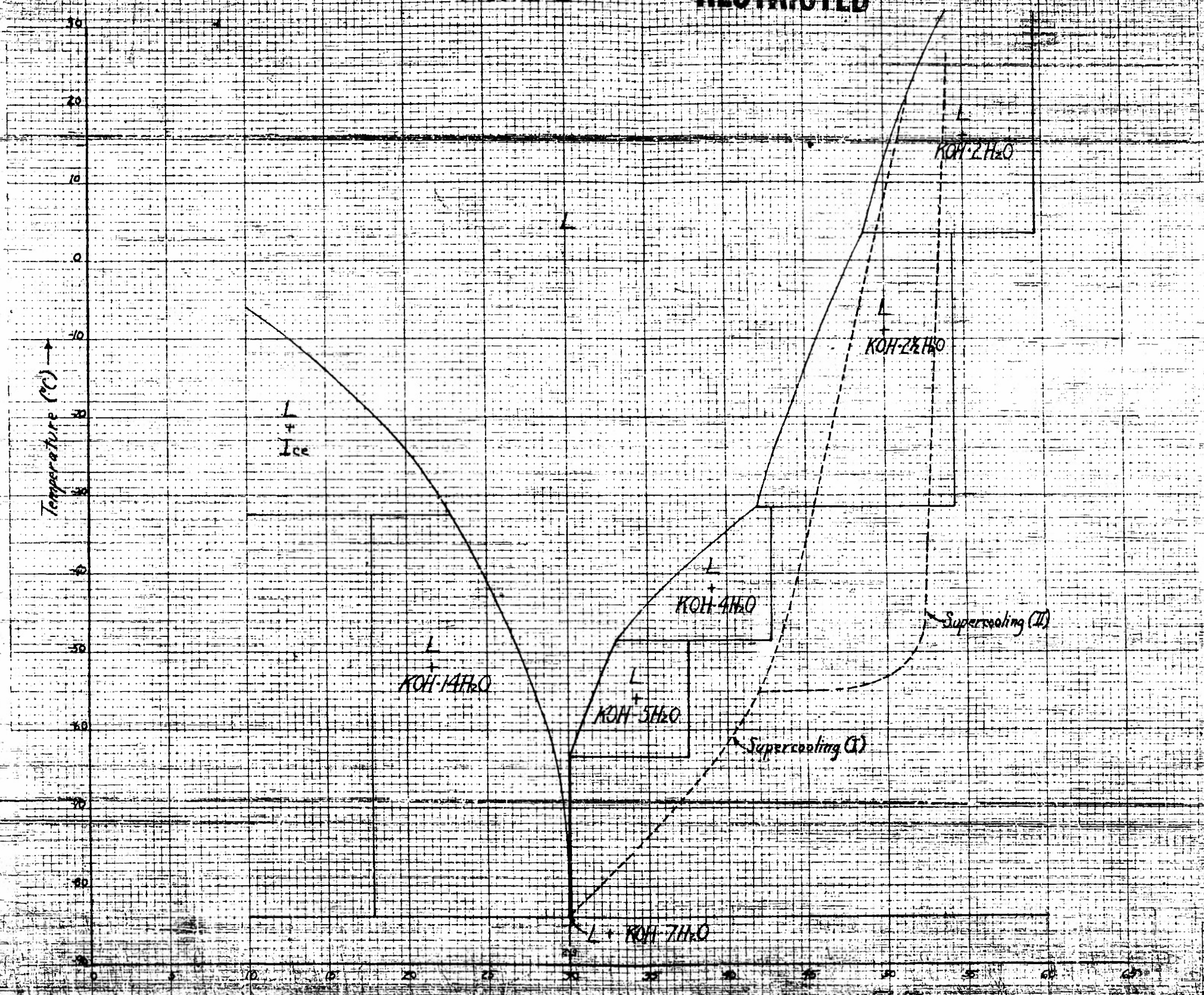
ANODE POLARIZATION IN MILLIVOLTS AT 0°C

Drain Rate..... 20 Milliamperes
Current Density..... 45.3 Ma. Sq. In.
Anode ..... .015" Zn Sheet
Amal. With Dil.
$Hg(CN)_2$ Solution

Hours Discharge	Polarization Values-Duplicating Tests 1	2	3	Average Value	Average to Nearest 5 Millivolts	Mean Value	Maximum Deviation From Mean Value
0	7.0	8.0	14.5	9.8	10	10.7	±3.7
½	7.2	7.2	12.0	8.8	10	9.6	±2.4
1	7.1	8.5	10.0	8.5	10	8.6	±1.5
1½	8.1	7.5	10.7	8.6	10	9.1	±1.6
2	9.0	9.5	11.0	9.8	10	10.0	±1.0
2½	10.5	7.8	11.0	9.8	10	9.4	±1.6
3	9.0	10.0	11.0	10.0	10	10.0	±1.0
3½	---	----	----	----	--	----	---
4	13.0	11.8	14.0	9.7	10	12.9	±1.1
4½	17.0	10.5	13.0	13.5	15	13.8	±3.3
5	18.8	10.0	15.0	14.6	15	14.2	±4.2
5½	17.8	10.7	13.5	14.0	15	14.2	±3.5
6	17.3	11.0	15.5	14.6	15	14.2	±3.2
6½	18.0	12.5	15.7	15.4	15	15.2	±2.7
7	18.0	11.7	----	----	--	----	---

KOH-ZNO(2.0%)-H_2O SYSTEM
PHASE DIAGRAM
FIGURE I

RESTRICTED



KOH-ZnO(4.0%)-H_2O SYSTEM
PHASE DIAGRAM
FIGURE II

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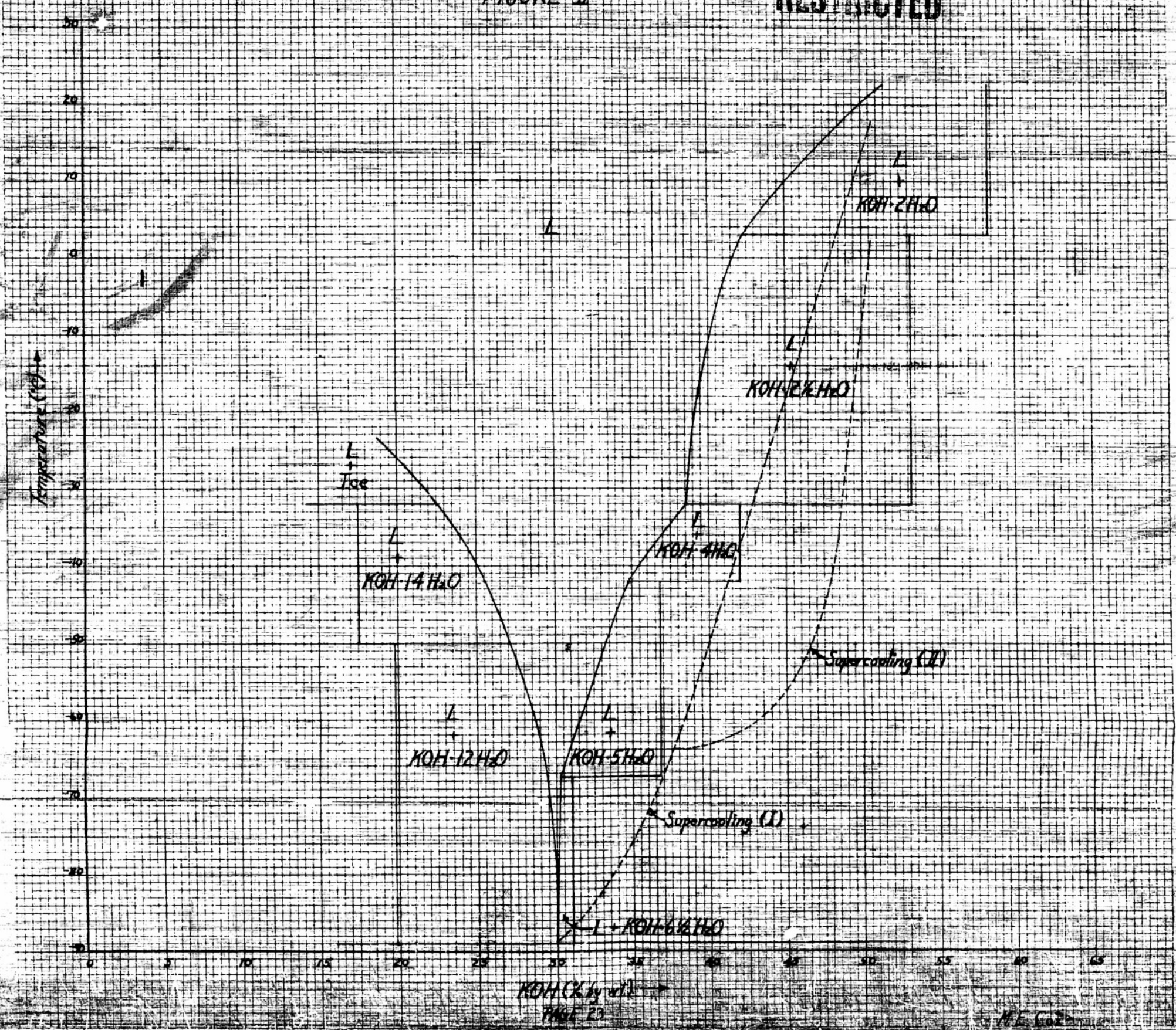
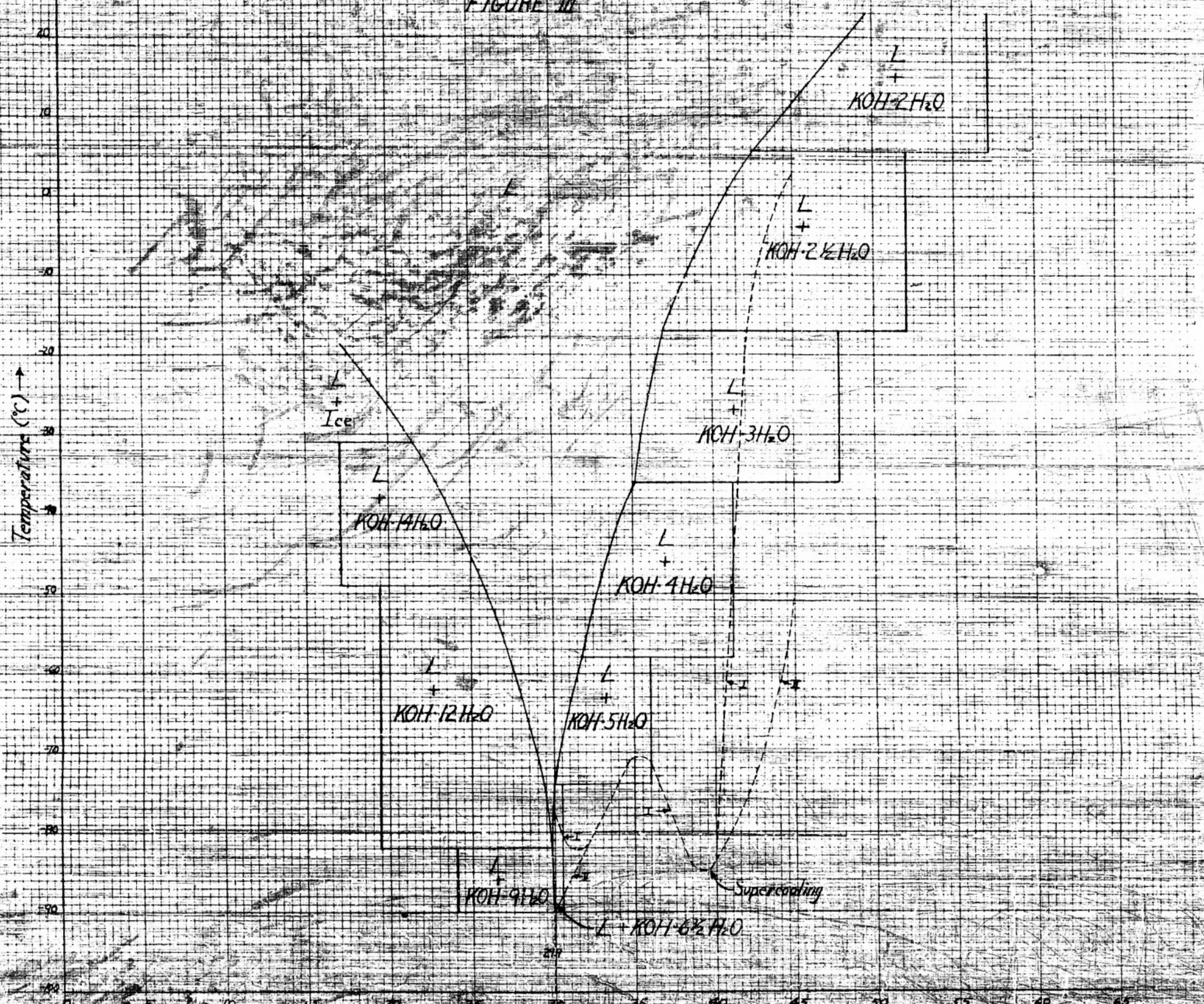


M.E. Cox

KOH-ZnO(6.0%)-H_2O SYSTEM

PHASE DIAGRAM

FIGURE III



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KOH-ZNO(18.0%)-H_2O SYSTEM
PHASE DIAGRAM
FIGURE III

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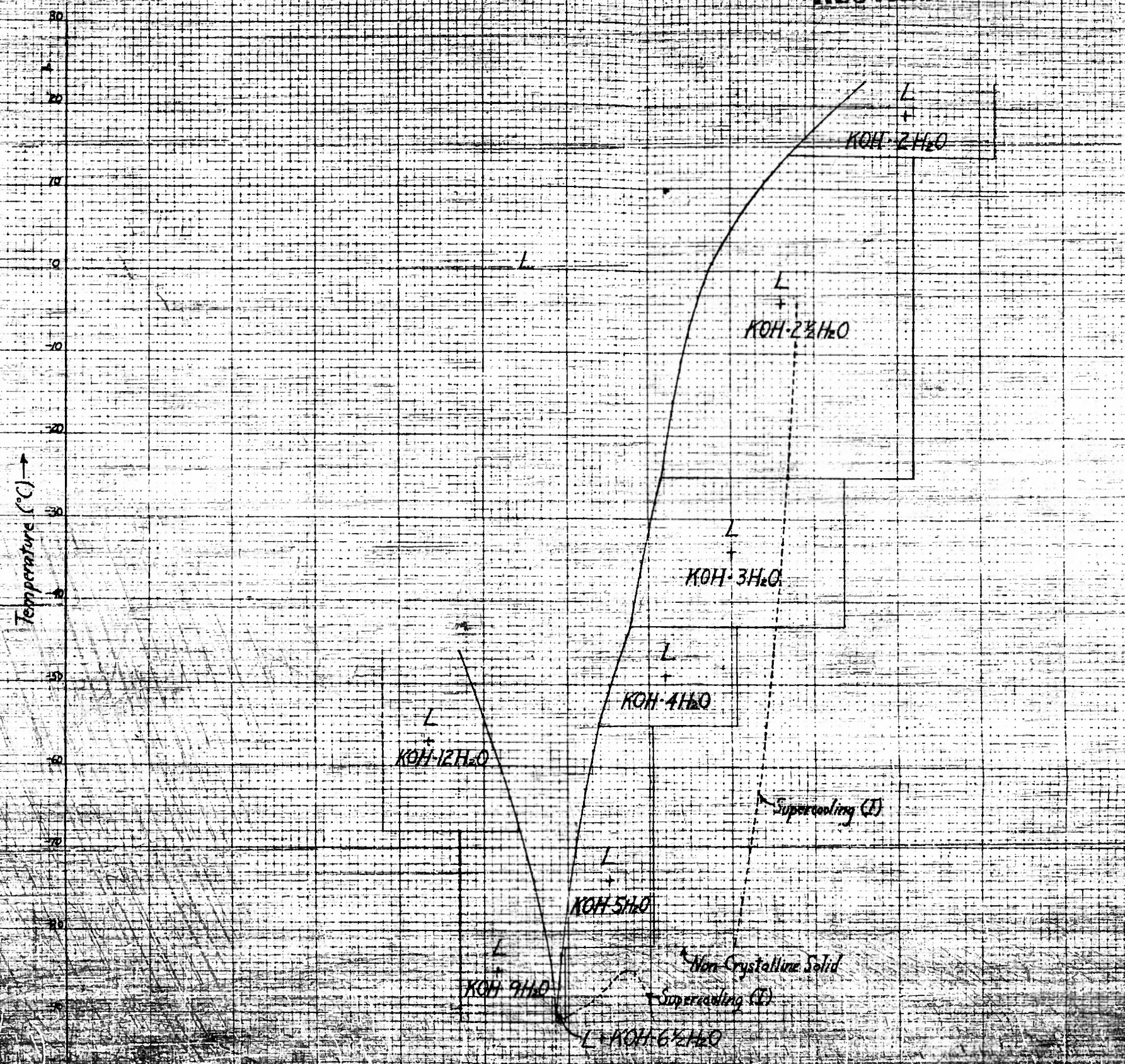

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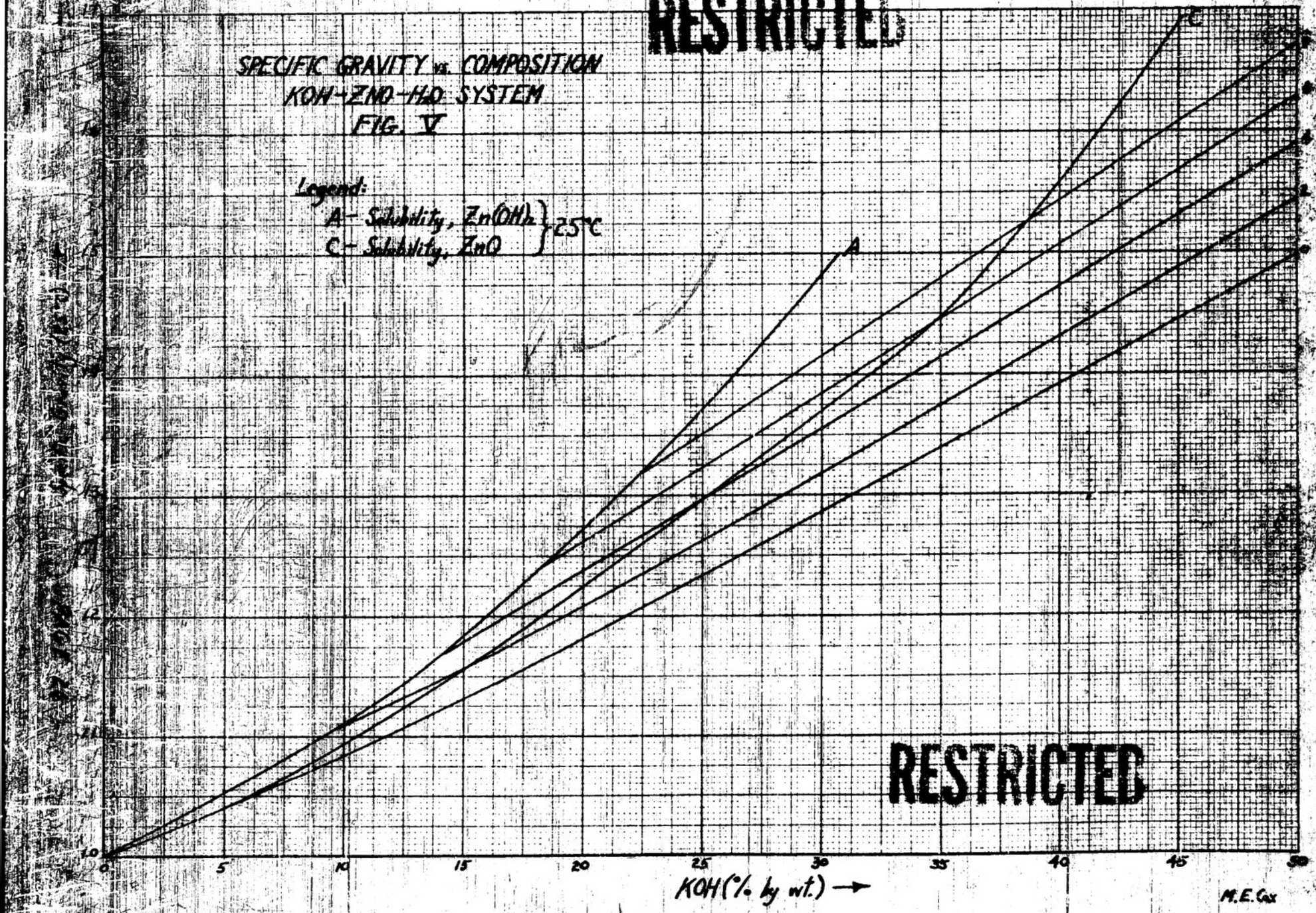

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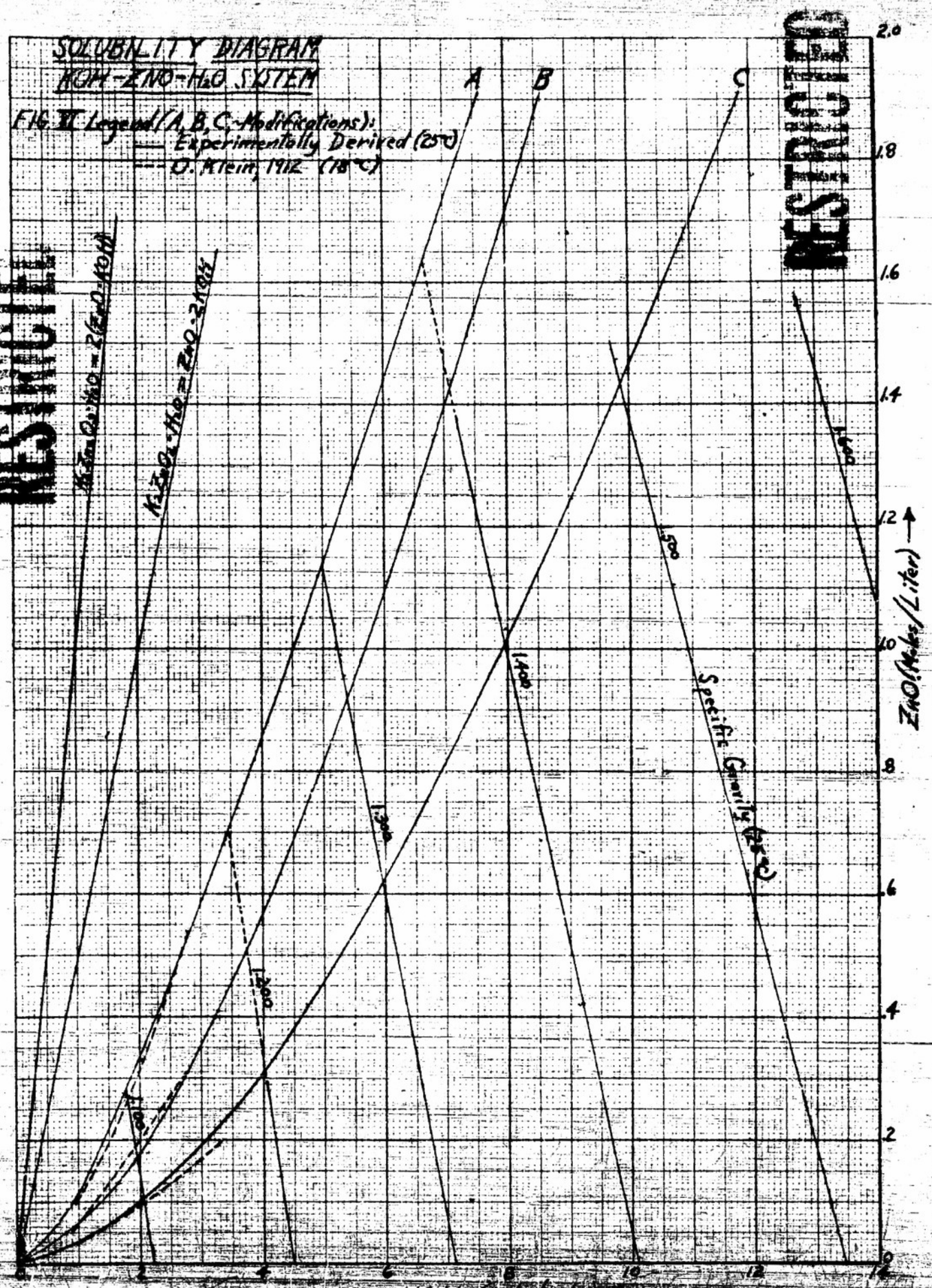

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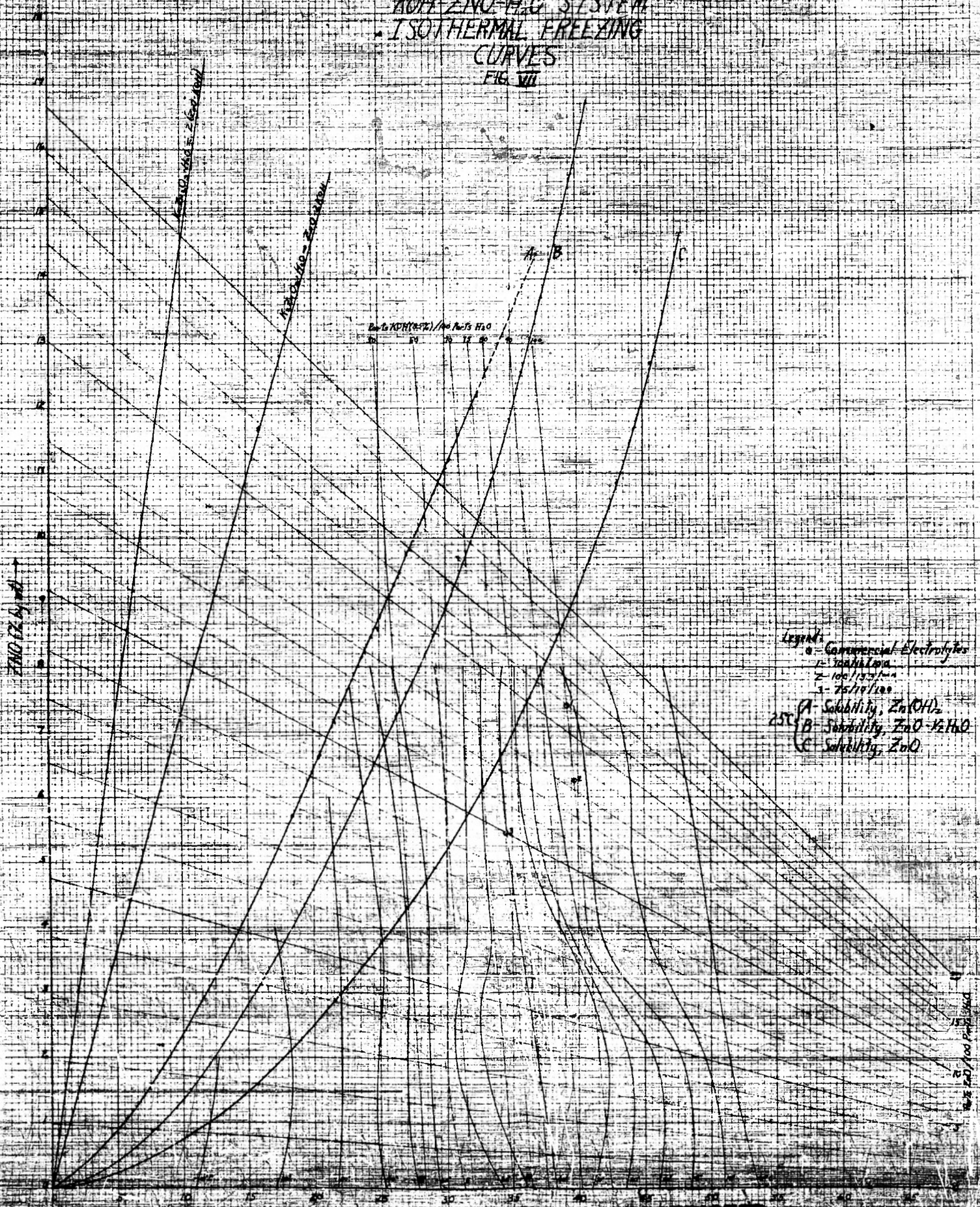
SOLUBILITY DIAGRAM KOH-ZNO-H_2O SYSTEM ISOTHERMAL FREEZING CURVES

FIG. VII



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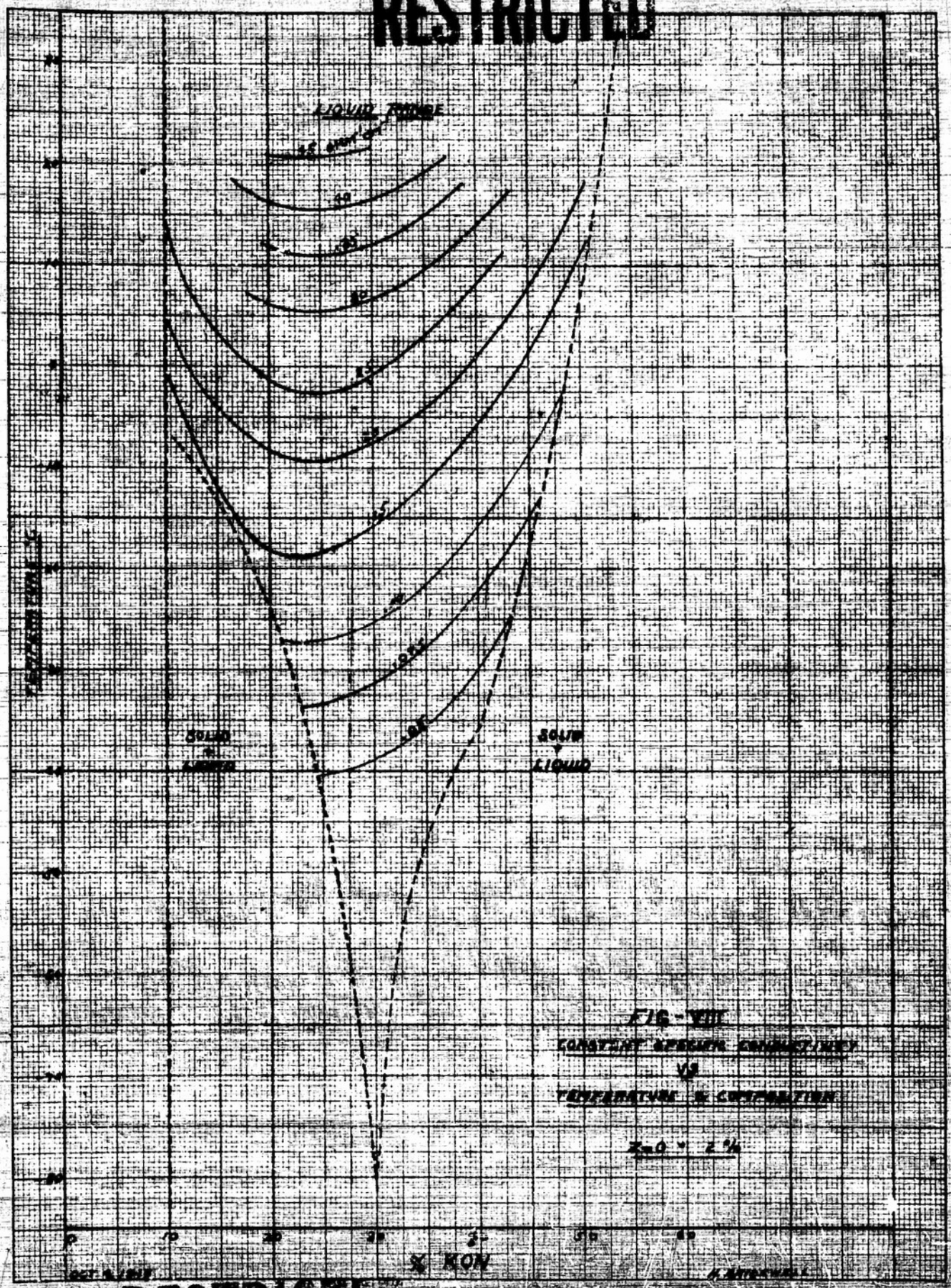

FIG - VIII
CONSTANT SPECIFIC CONDUCTIVITY
VS
TEMPERATURE & COMPOSITION
Z=0 - 2%

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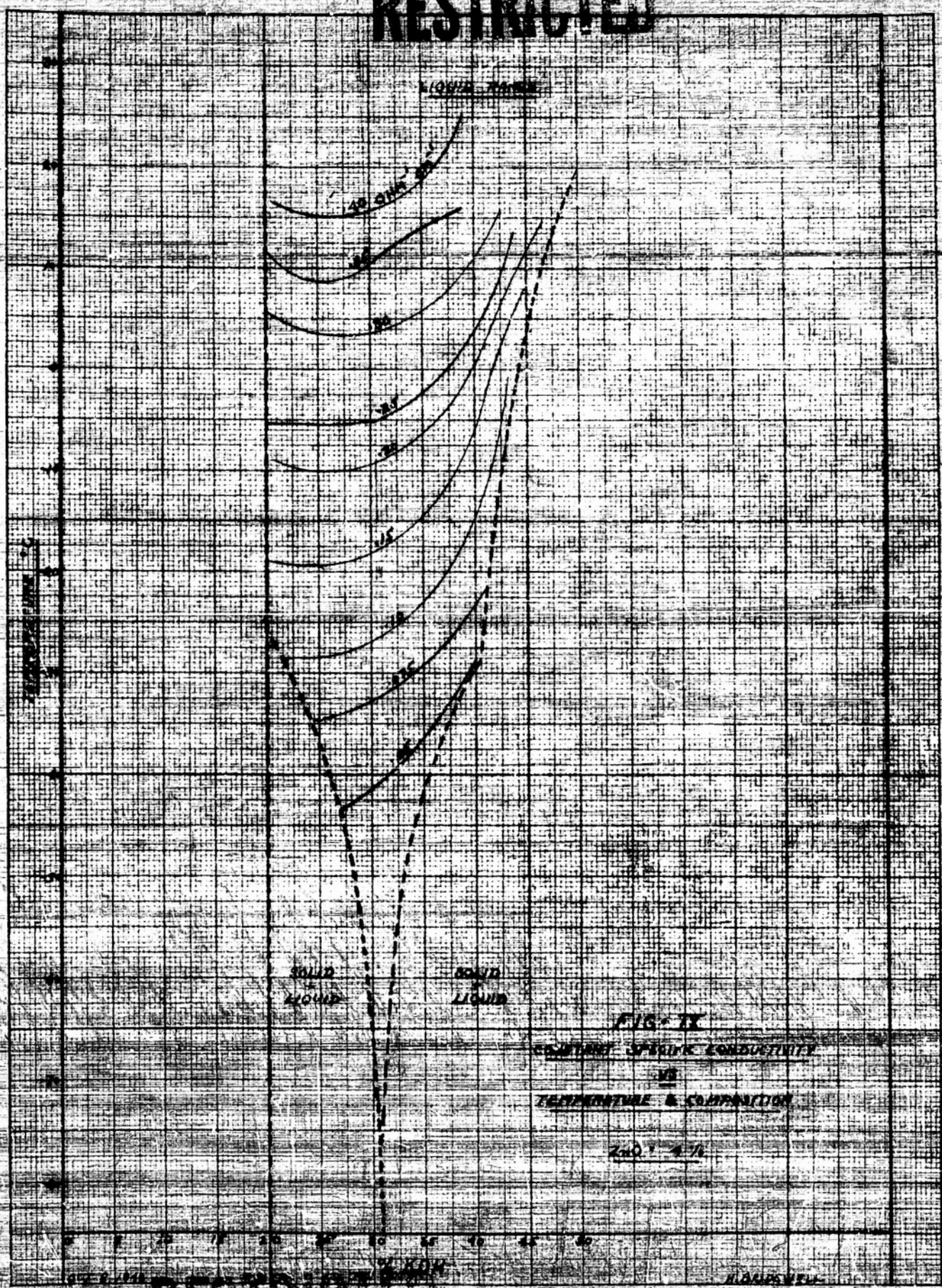

FIG. IX

EQUIVALENT SPECIFIC CONDUCTIVITY

VS

TEMPERATURE & COMPOSITION

ZnO = 4 %

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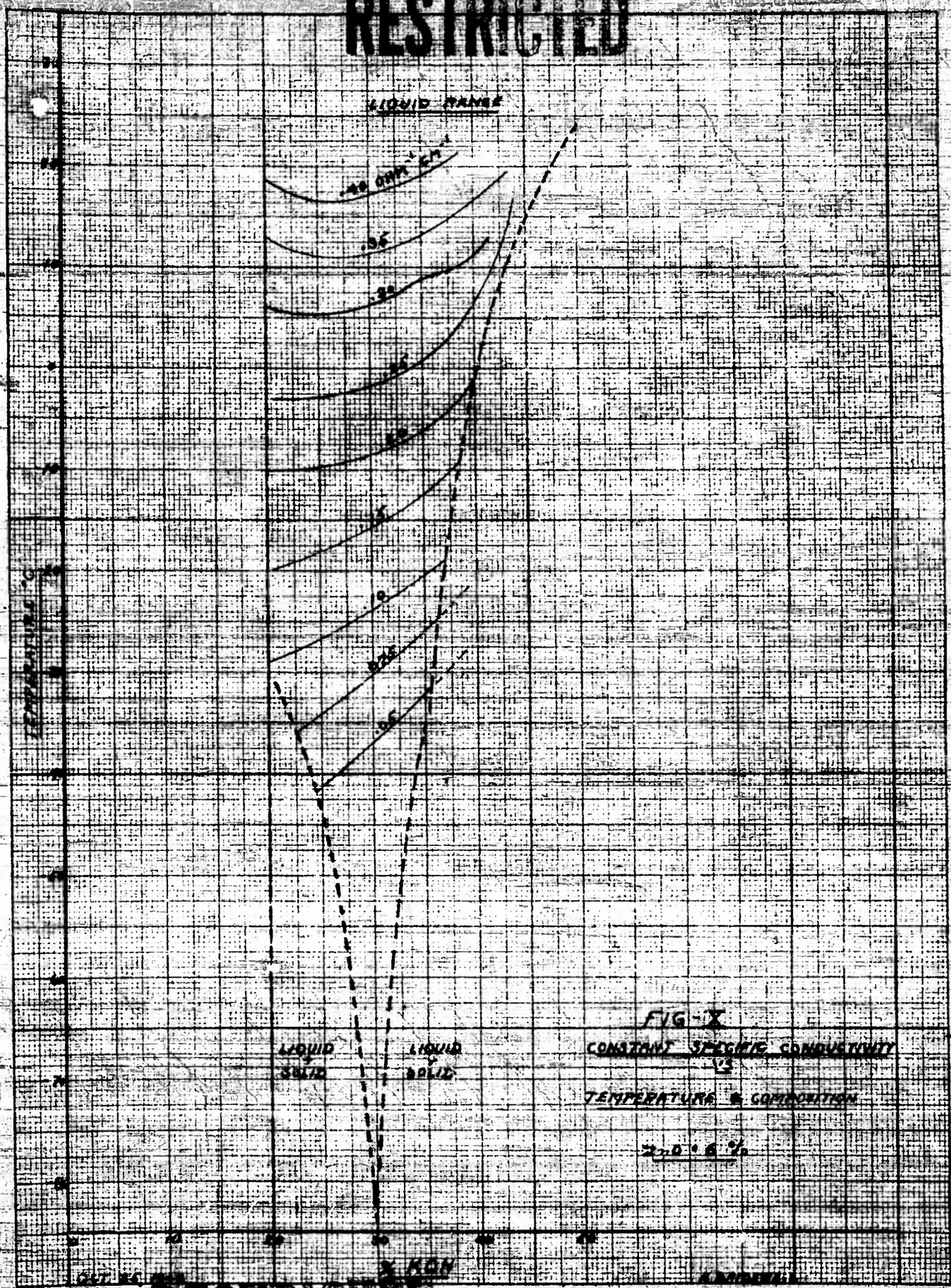

FIG - X

CONSTANT SPECIFIC CONDUCTIVITY

TEMPERATURE & COMPOSITION

2.0 - 6 %

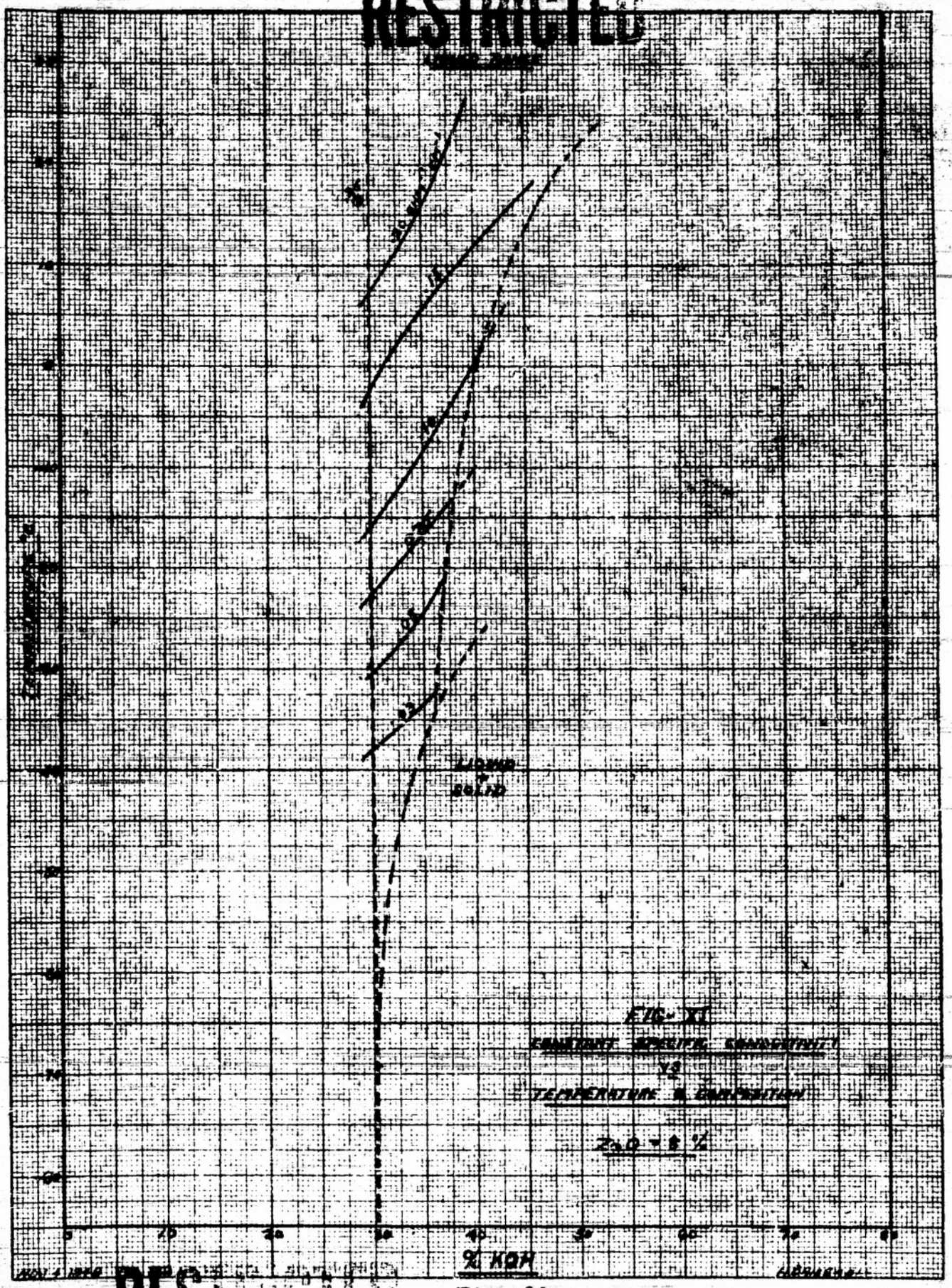

FIG. XI
CONSTANT SPECIFIC CONDUCTIVITY
VS
TEMPERATURE & COMPOSITION
Z_2O = 8 %

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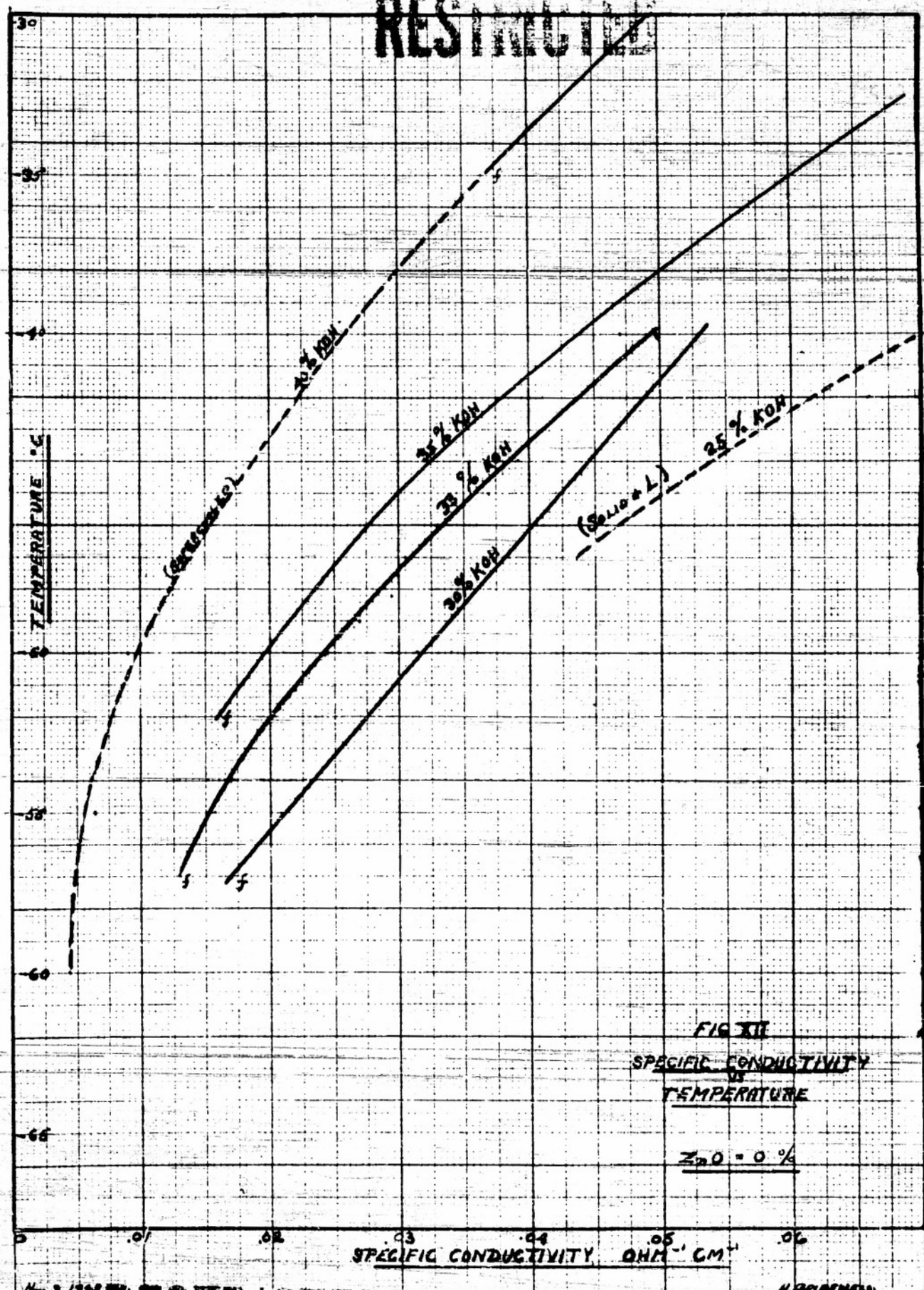

FIG XII
SPECIFIC CONDUCTIVITY vs TEMPERATURE

$ZnO = 0\%$

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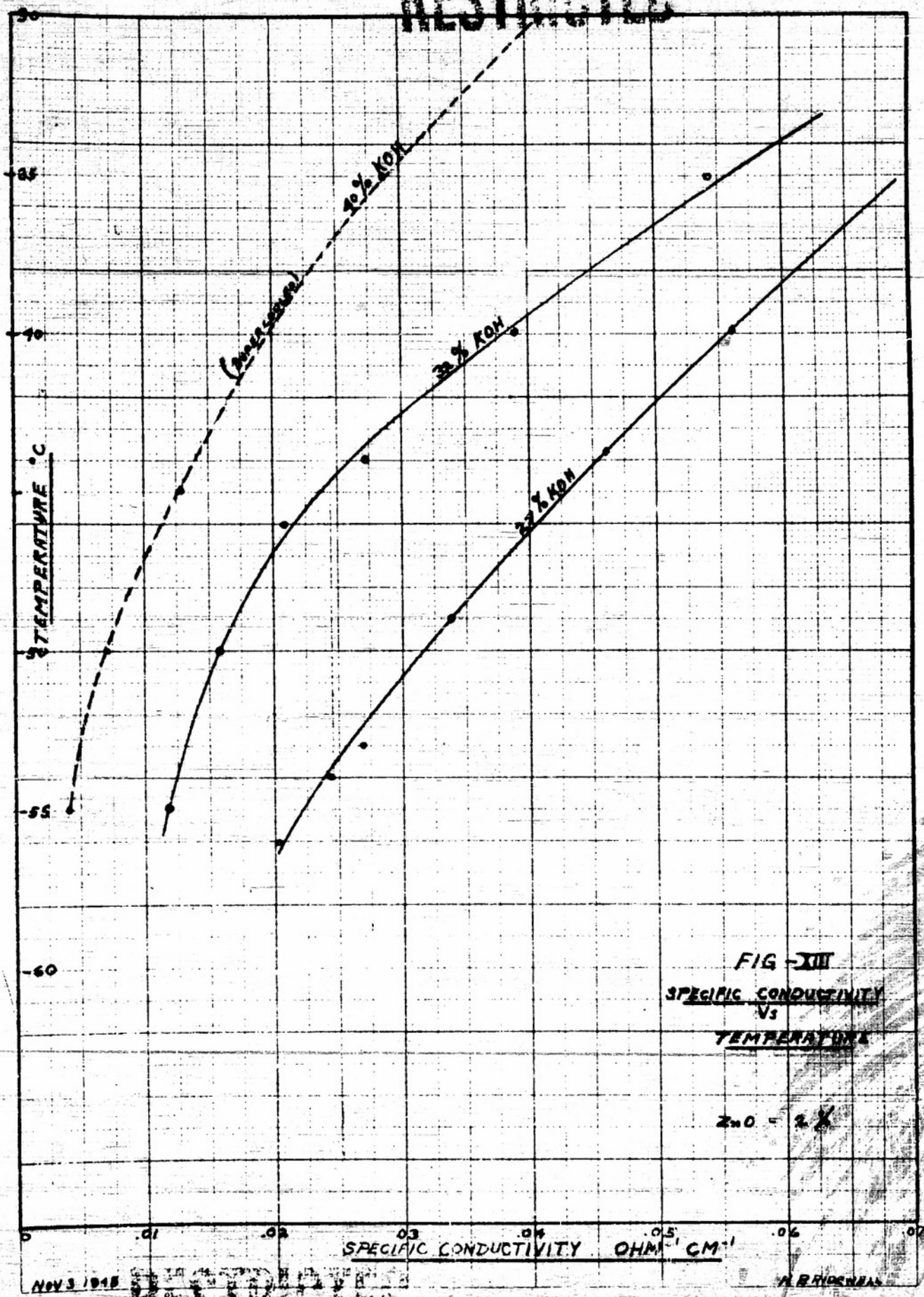

FIG - XIII

SPECIFIC CONDUCTIVITY Vs TEMPERATURE

ZnO = 2%

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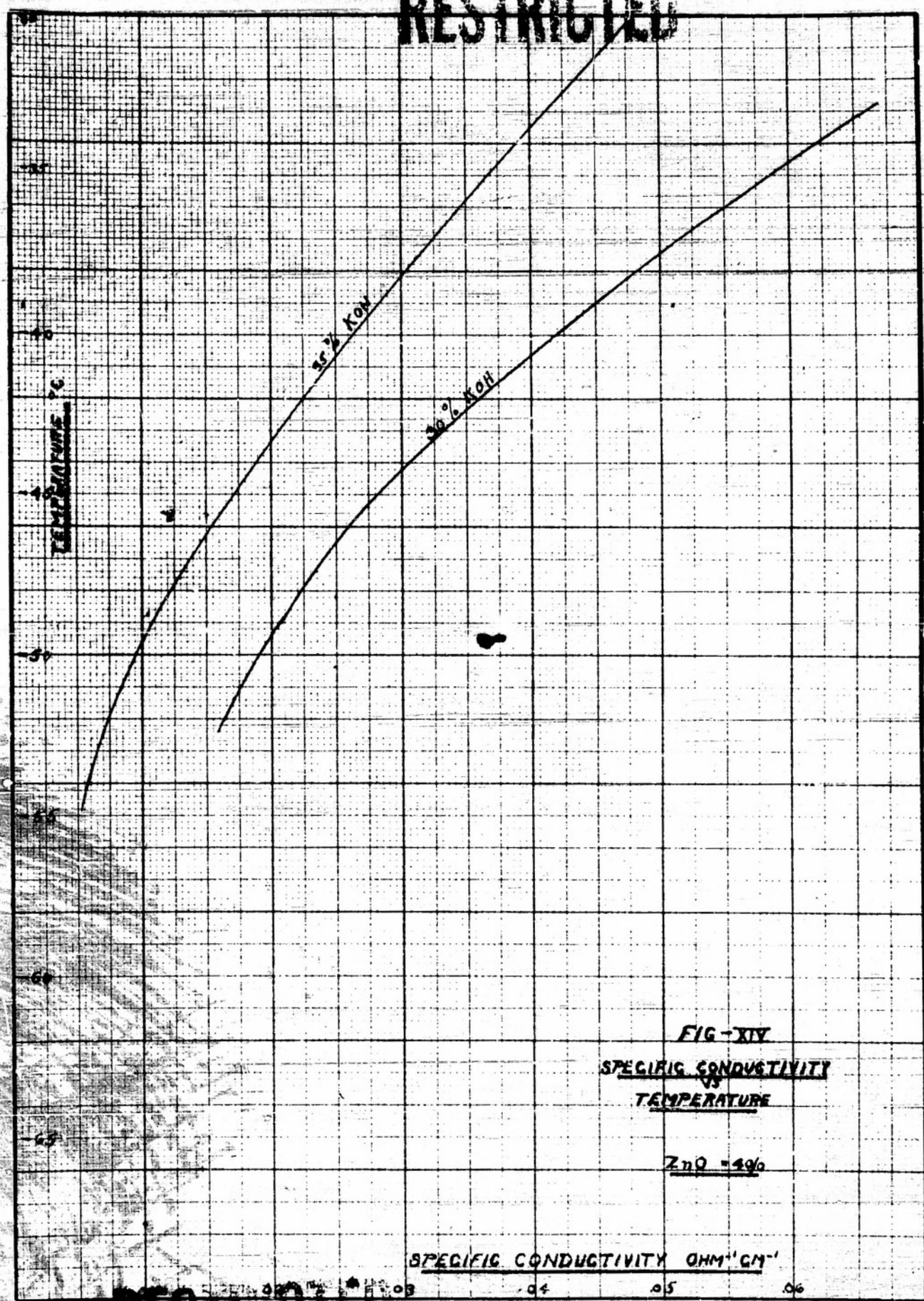

FIG - XIV
SPECIFIC CONDUCTIVITY
vs
TEMPERATURE

ZnO = 4%

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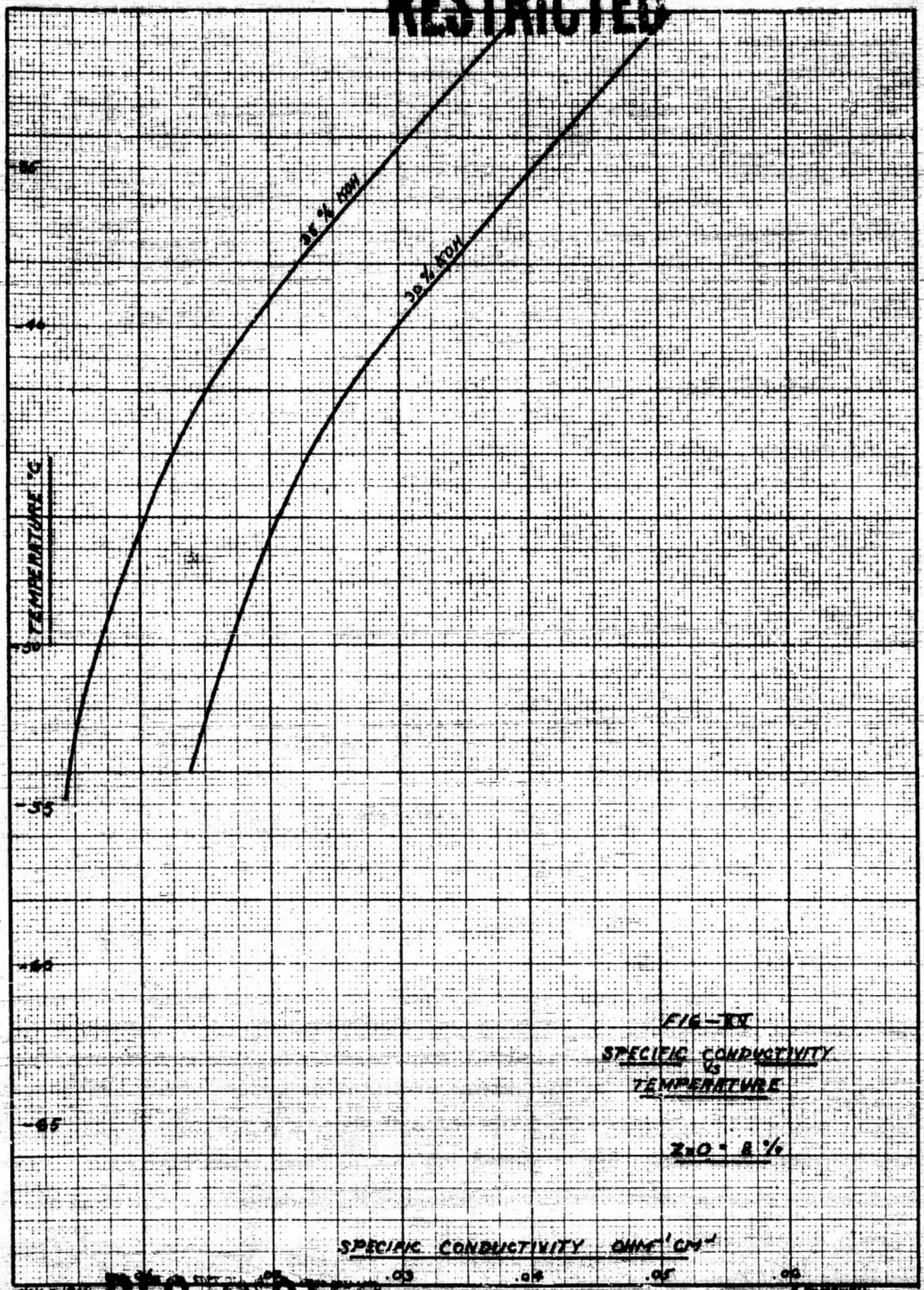

FIG-XII
SPECIFIC CONDUCTIVITY vs TEMPERATURE
ZnO = 8 %

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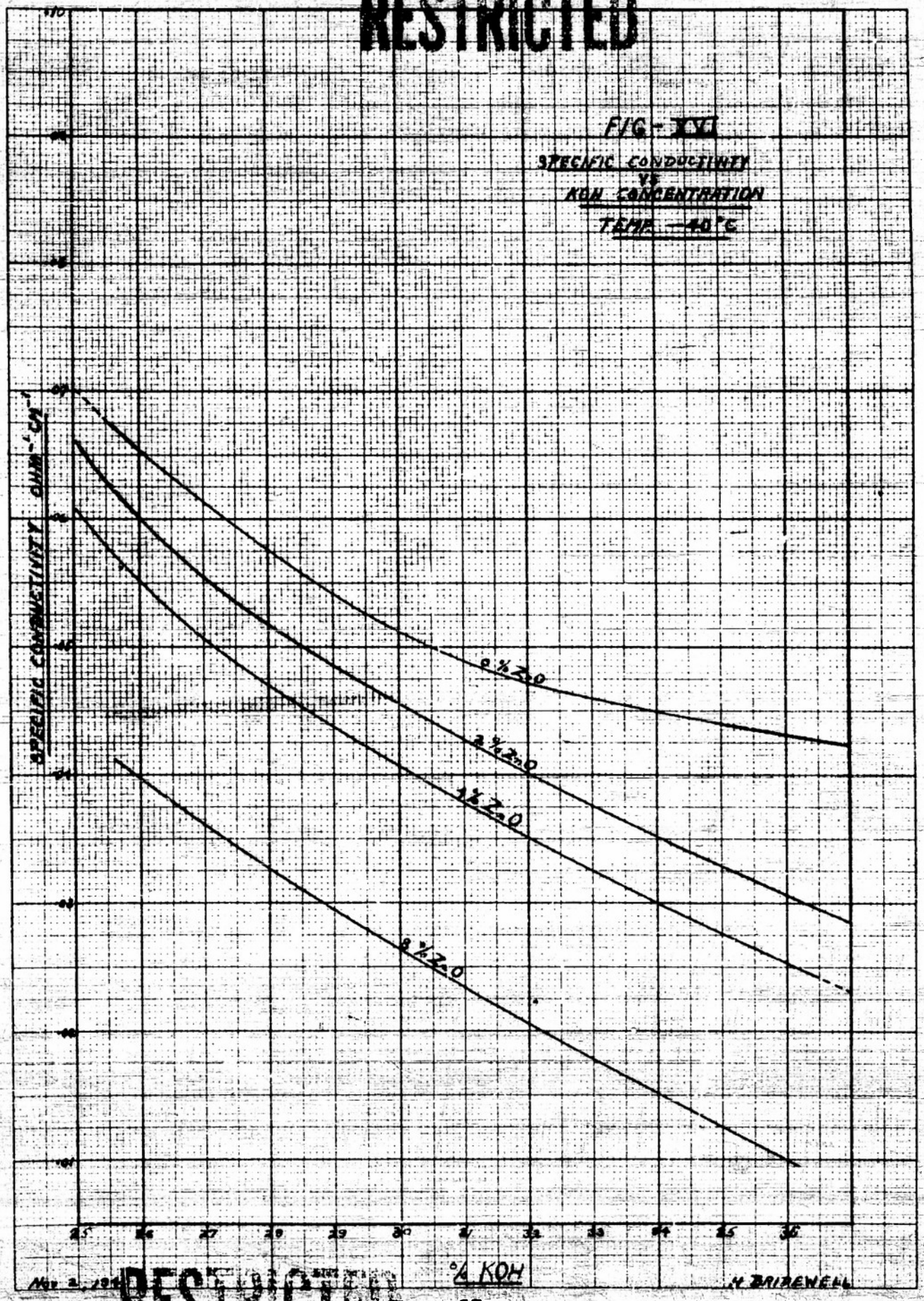

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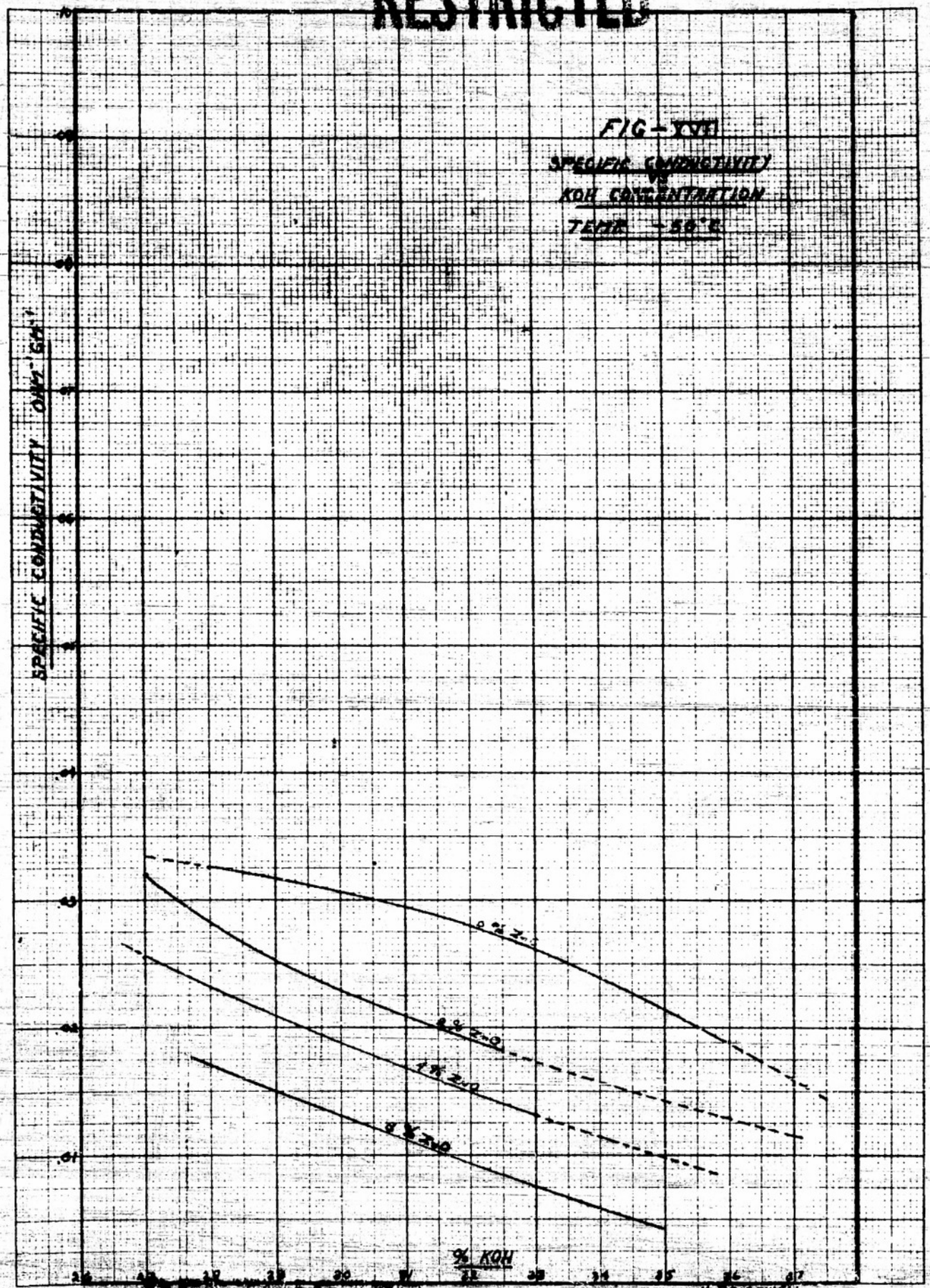

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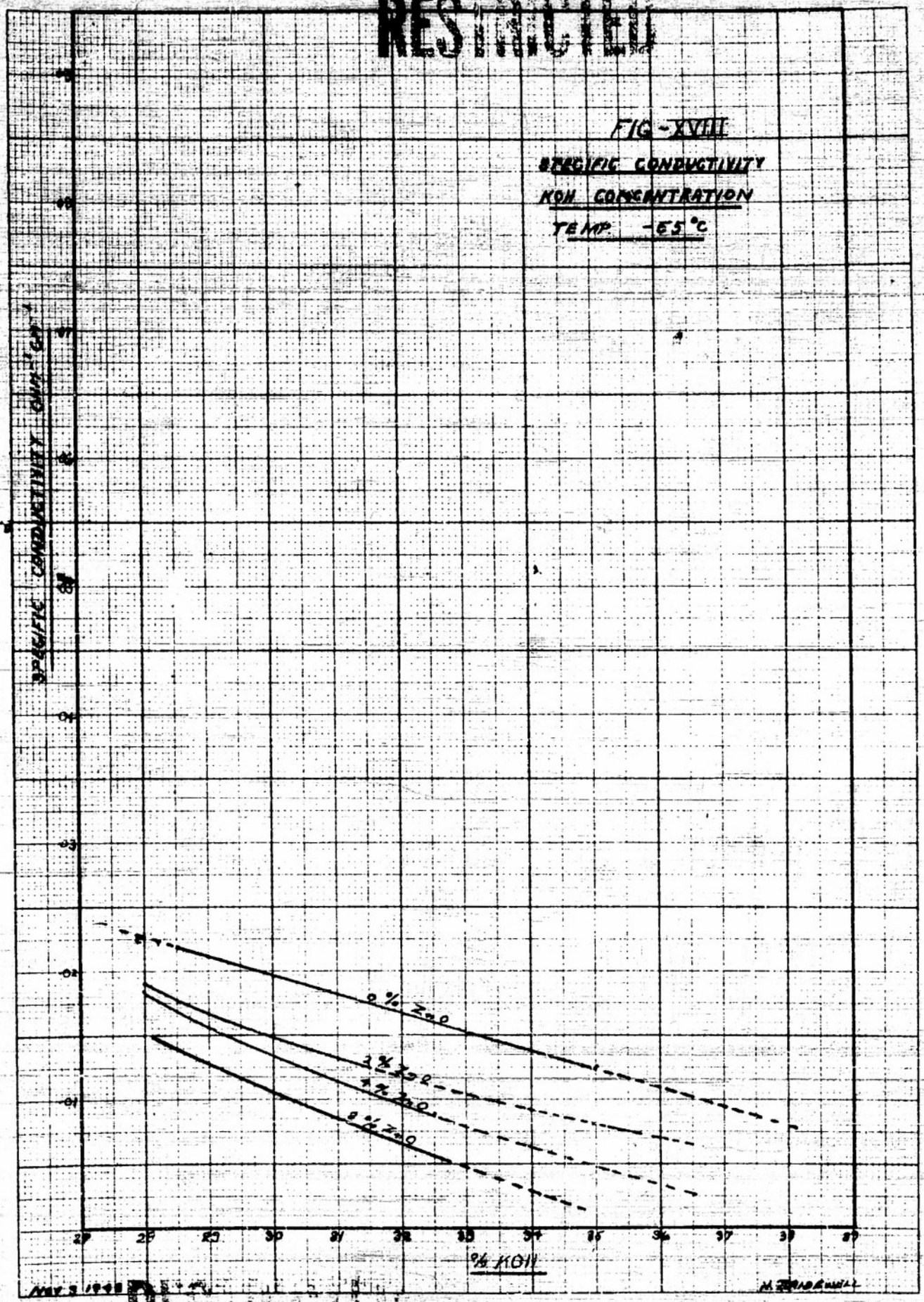

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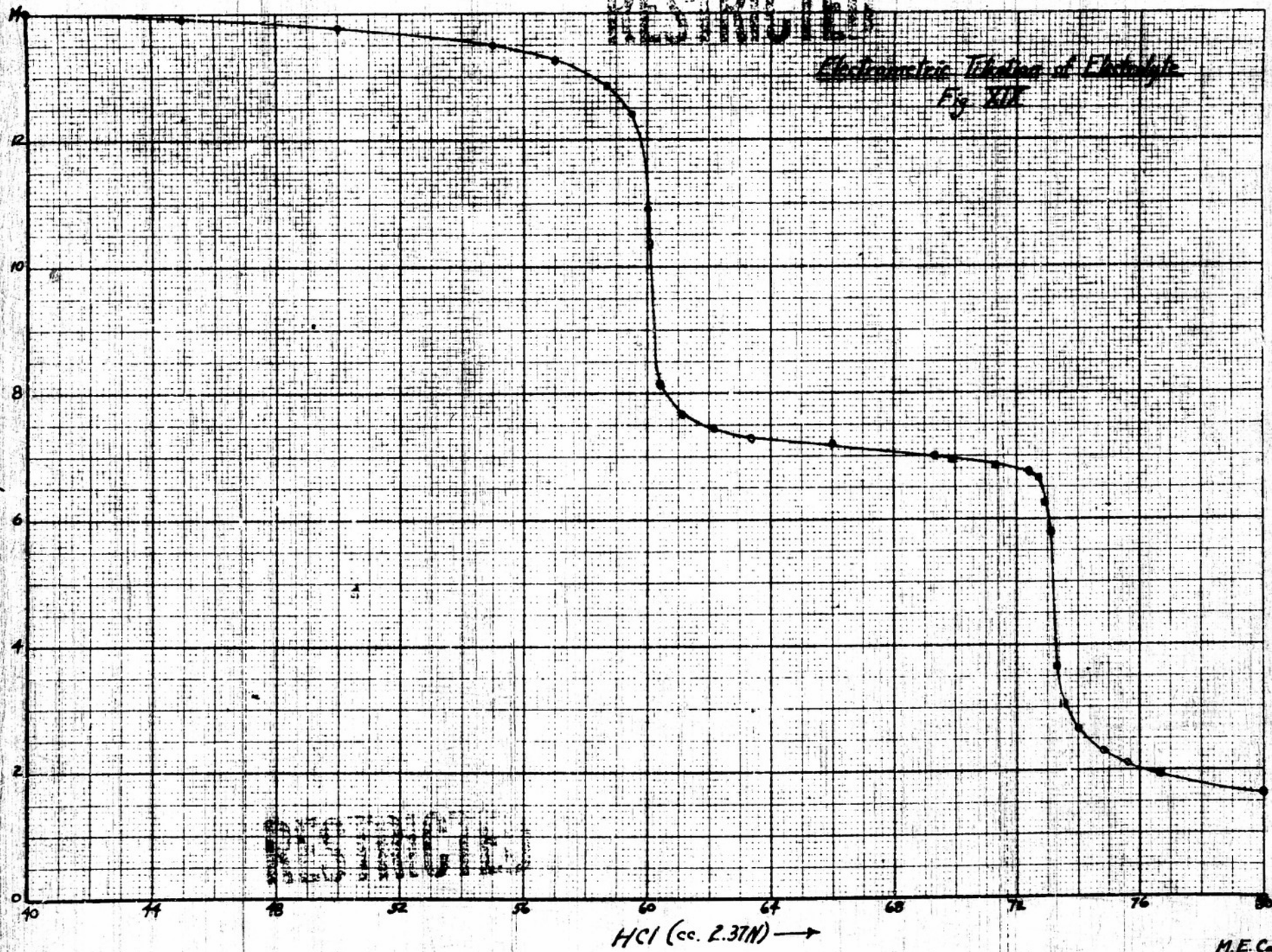
Electrometric Titration of Electrolyte
Fig. XII



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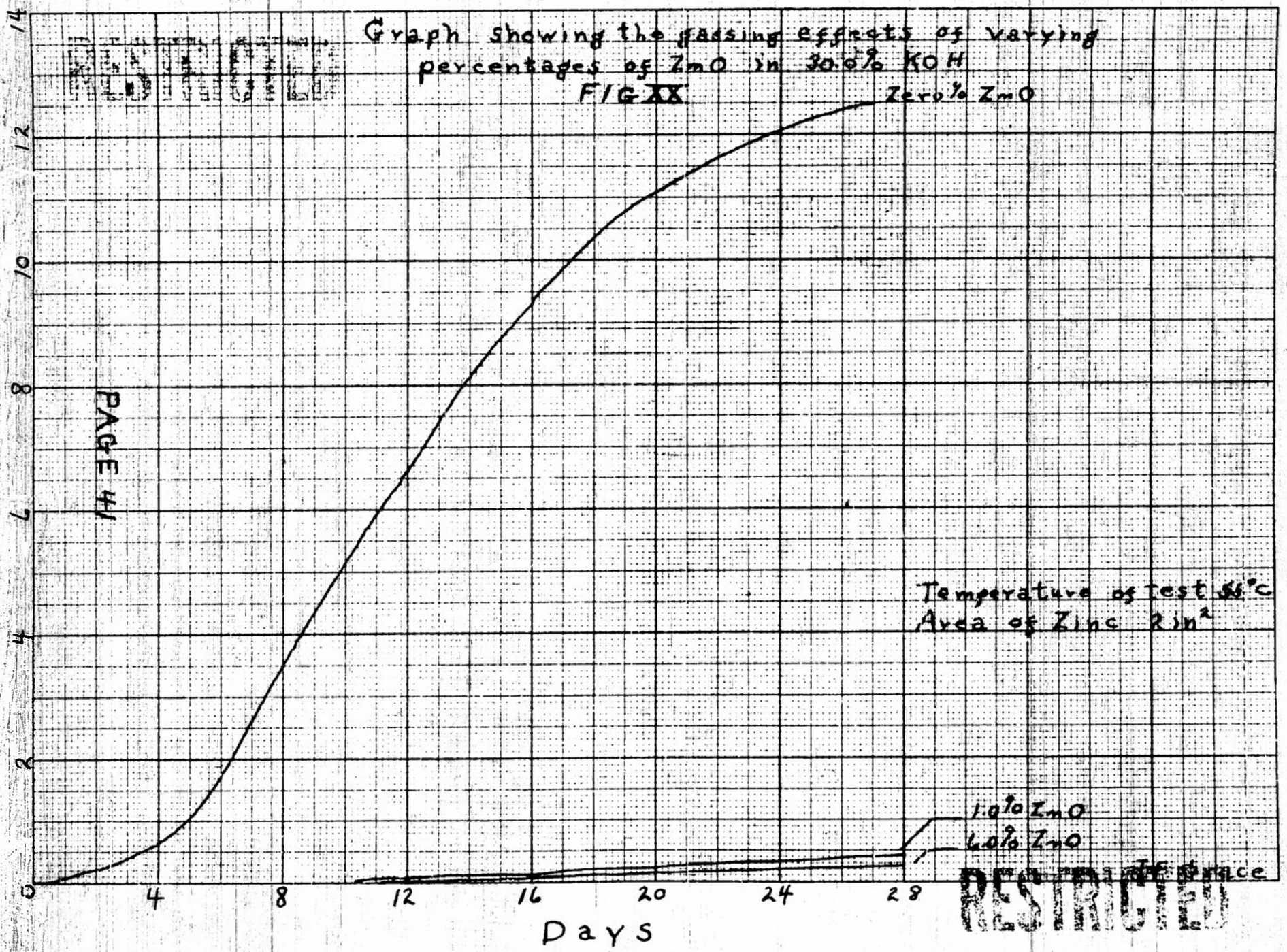

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RM-1R CELLS WITH $Ag_2O \cdot C$ (92.8) DEPOLARIZER

FIG. XXI

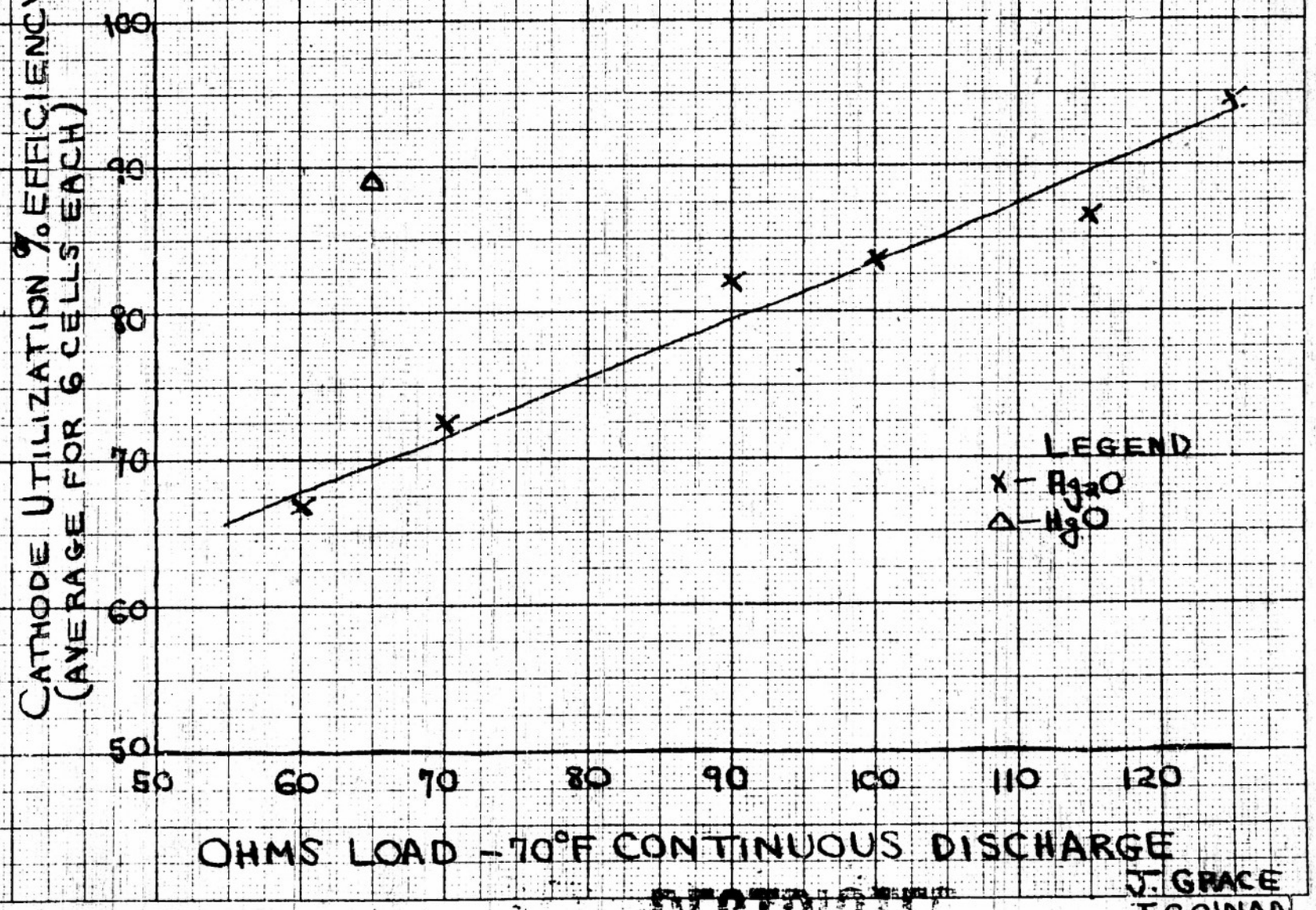

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FIGURE XXII

(LEGEND)

DETAIL OF END PLATE MODIFICATION

A. Haring Cell Body

B. Neoprene Sealing Gasket

C. Lucite Sleeve

D. End Plate

E. Depolarizer Wall

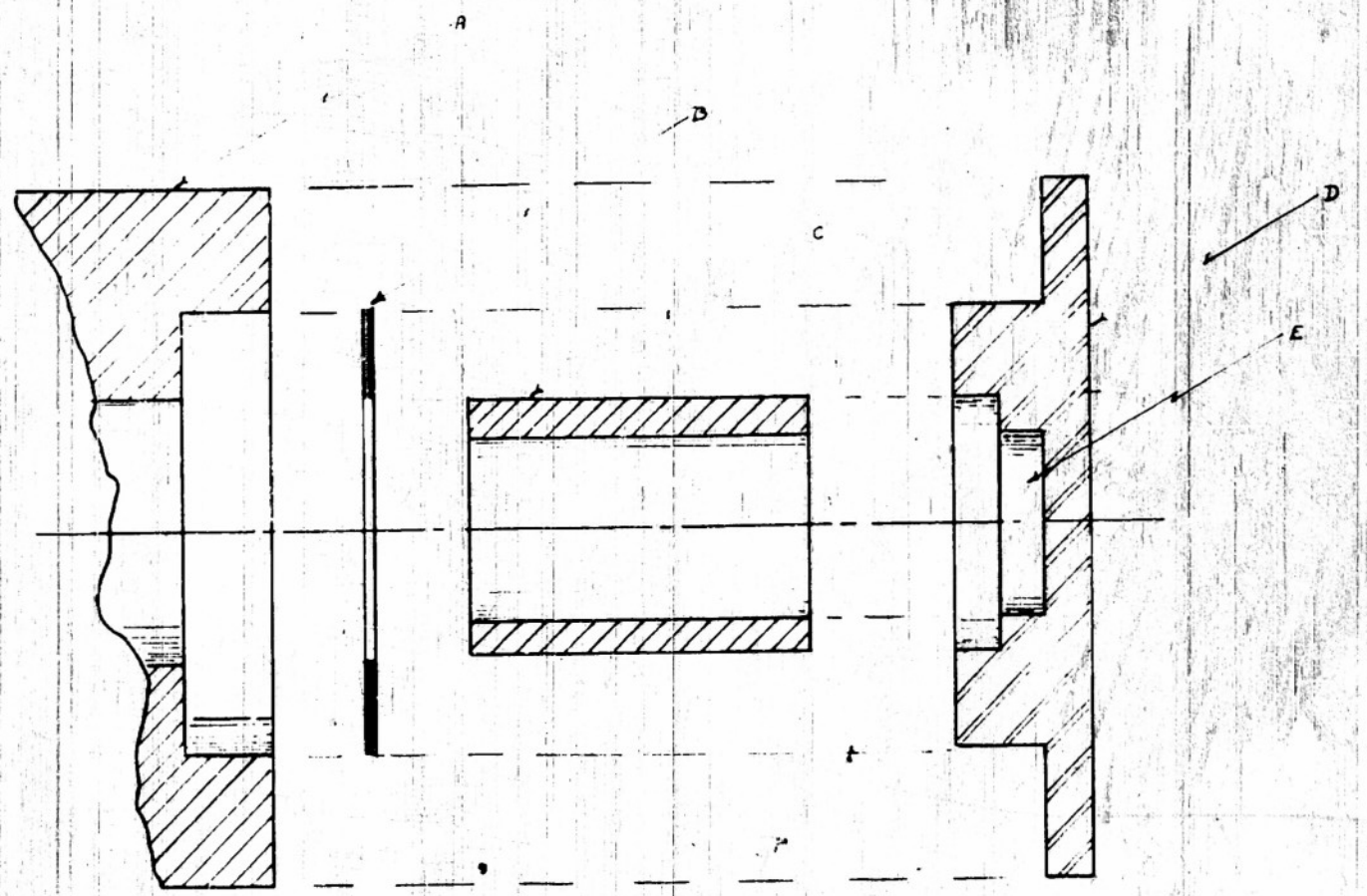

FIG - XXII

DETAIL OF END-PLATE MODIFICATION

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PER INCH
EUGENE DIETZGEN CO.

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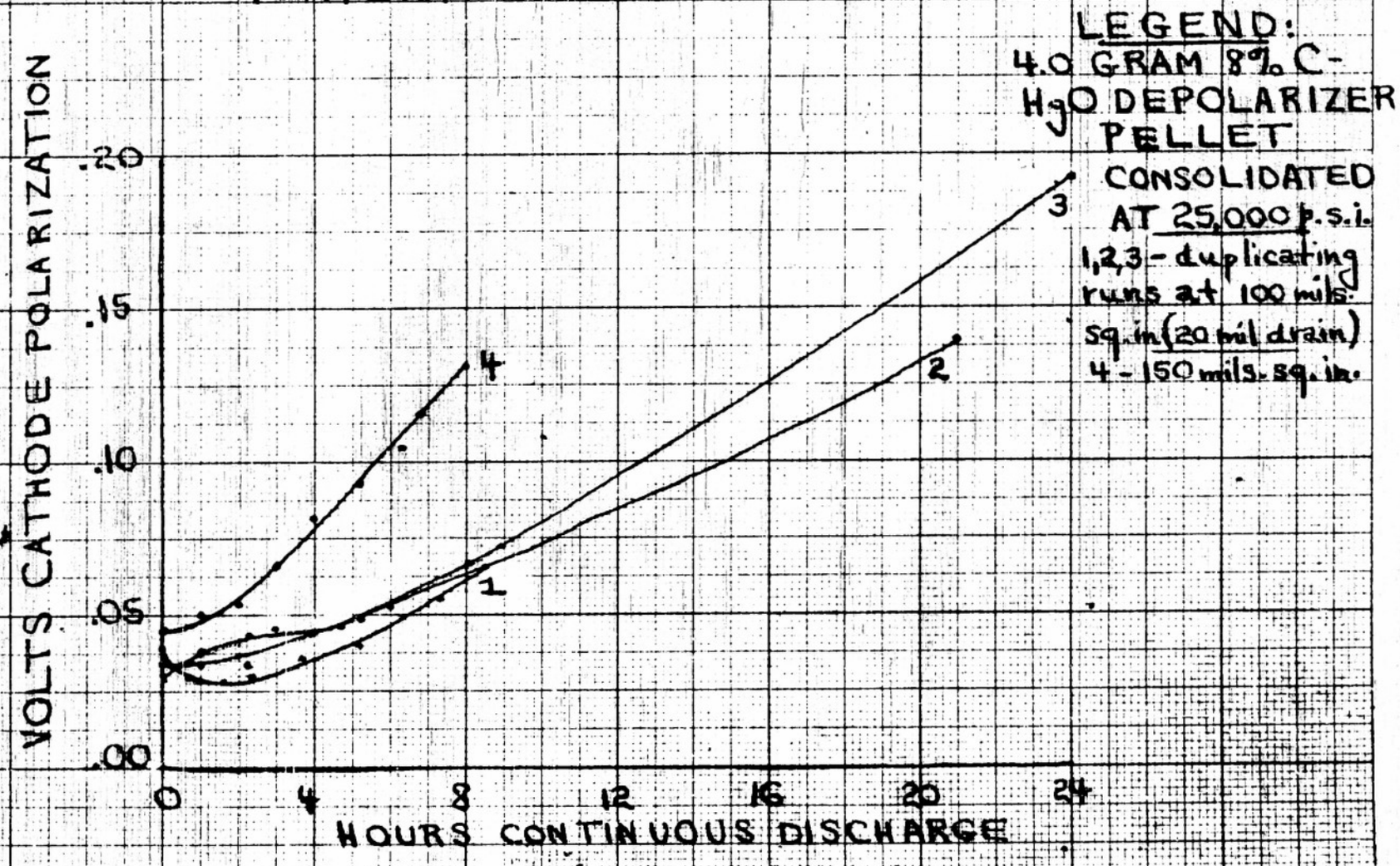
ROOM TEMPERATURE TESTS
WITH 0.5 IN. DIAMETER HARING CELL
FIG. XXIII
TEMPERATURE RANGE 23-29°C



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EUGENE DIETZGEN CO.

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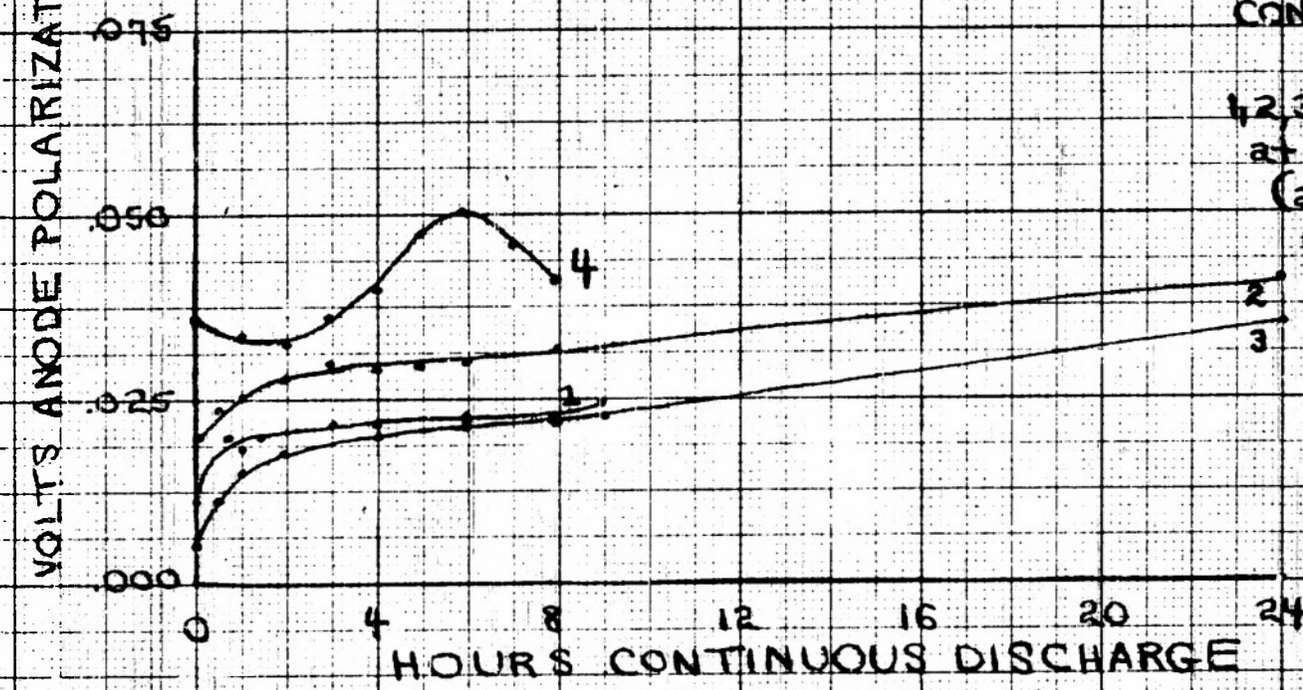
ROOM TEMPERATURE TESTS
WITH 0.5 IN. DIAMETER HARING CELL
FIG. XXIV
TEMPERATURE RANGE 23-29°C



LEGEND:
3.0 GRAM 13% Hg
AMAL. Zn POWDER
PELLET
CONSOLIDATED AT
6000 p.s.i.
1, 2, 3 - duplicating runs
at 100 mils. sq. in.
(20 mil drain)
4 - 150 mils. sq. in.

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FIGURE XXV

(LEGEND)

MODIFIED HARING CELL - GENERAL DETAIL

A. Steel End Plate and Electrode Holder
$1^{7}/_{8}$" O.D. $1\frac{1}{4}$" I.D. Electrode Compartment $\frac{3}{4}$" I.D.

B. Cathode - Compressed Mercuric Oxide - Graphite Depolarizer

C. Neoprene Sealing Gasket

D. Removable Reference Electrodes

E. Center Shaft - Teflon

F. Thermometer Well

G. Haring Cell Body - Teflon - $2\frac{3}{4}$" long, $1^{7}/_{8}$"O.D., $\frac{3}{4}$"I.D.

H. Neoprene Sealing Gasket

I. Anode - .025" Zinc Disc

J. Copper End Plate and Electrode Holder

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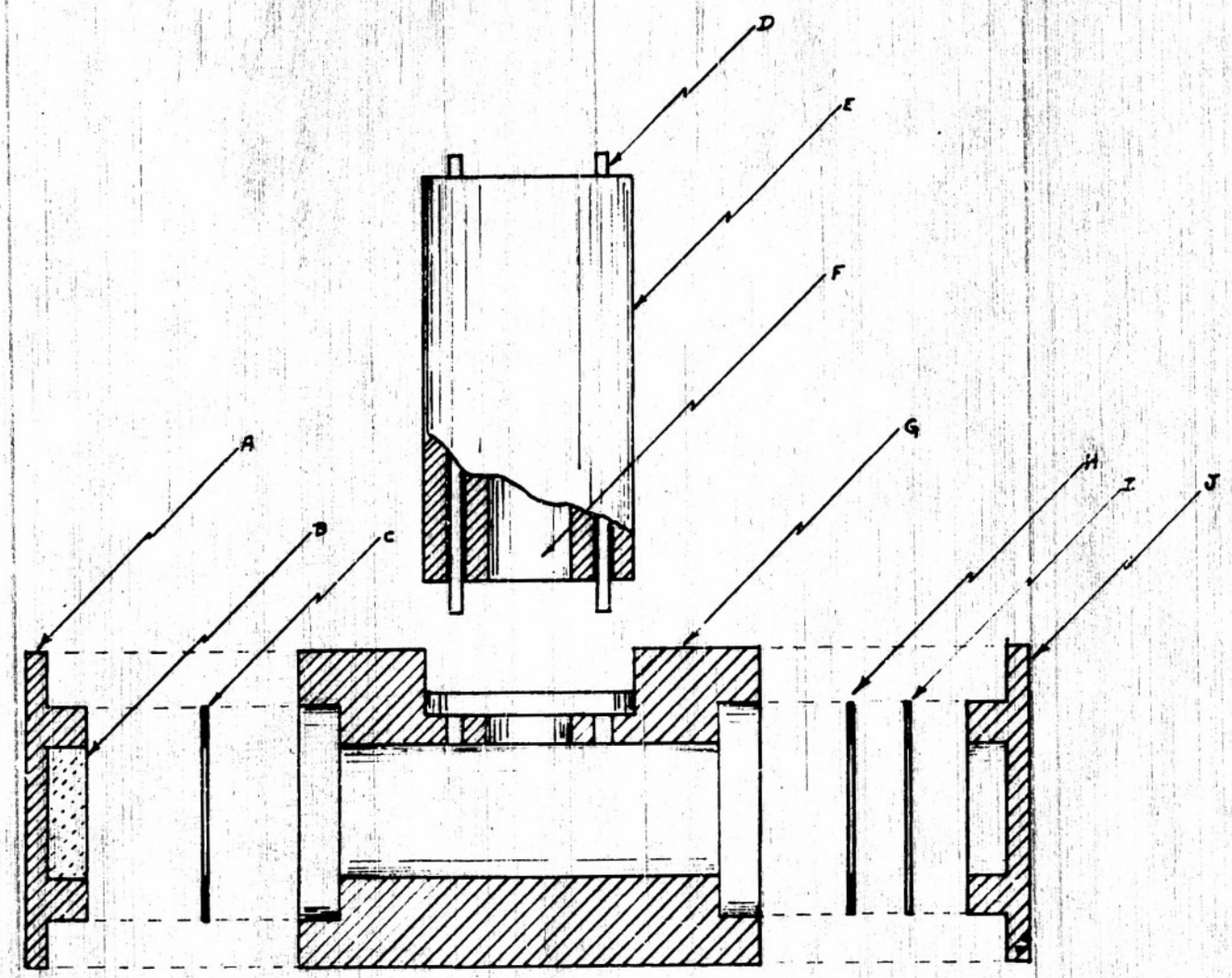

FIG - XXV

MODIFIED HARING CELL

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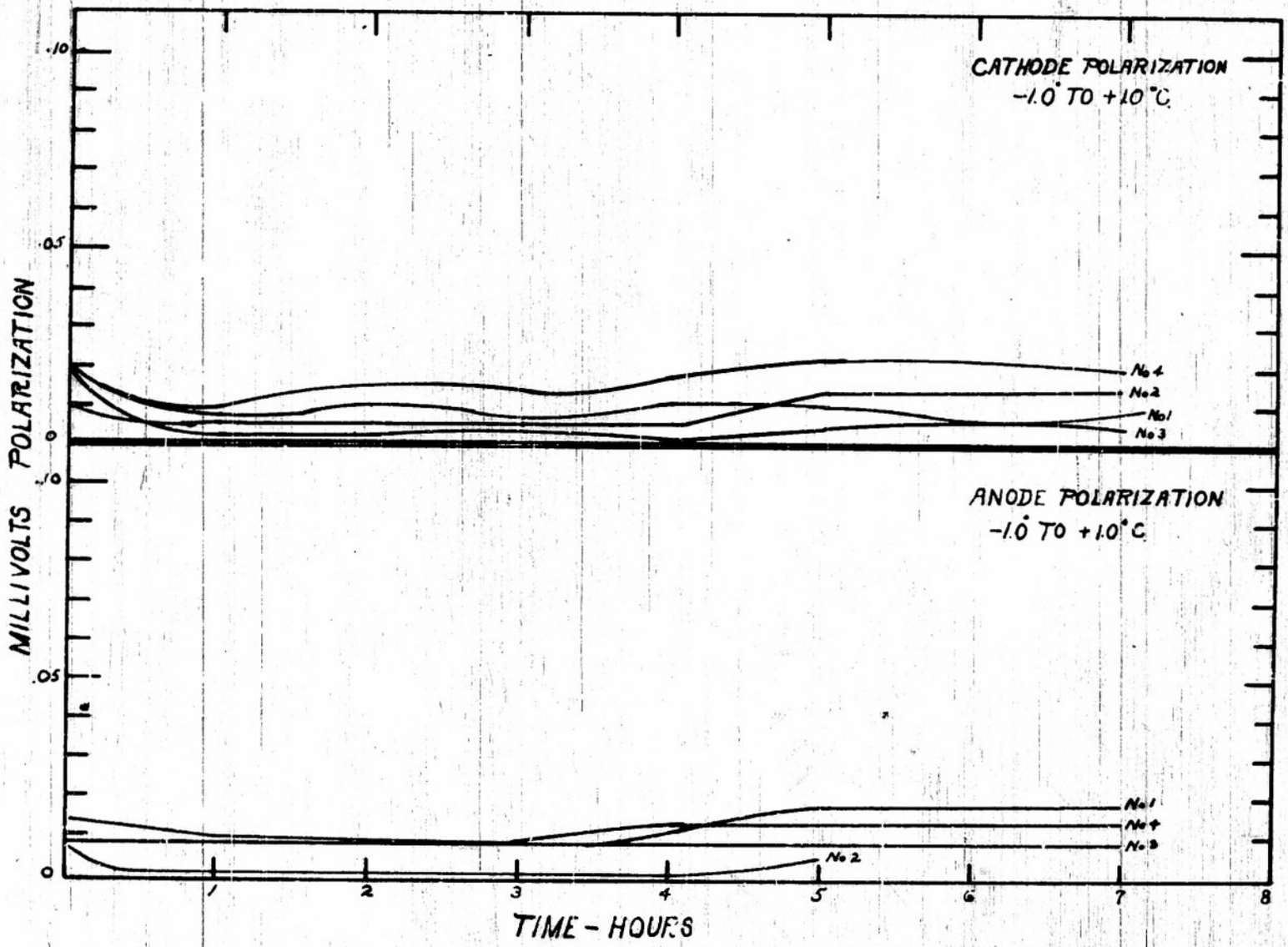

FIG XXVI

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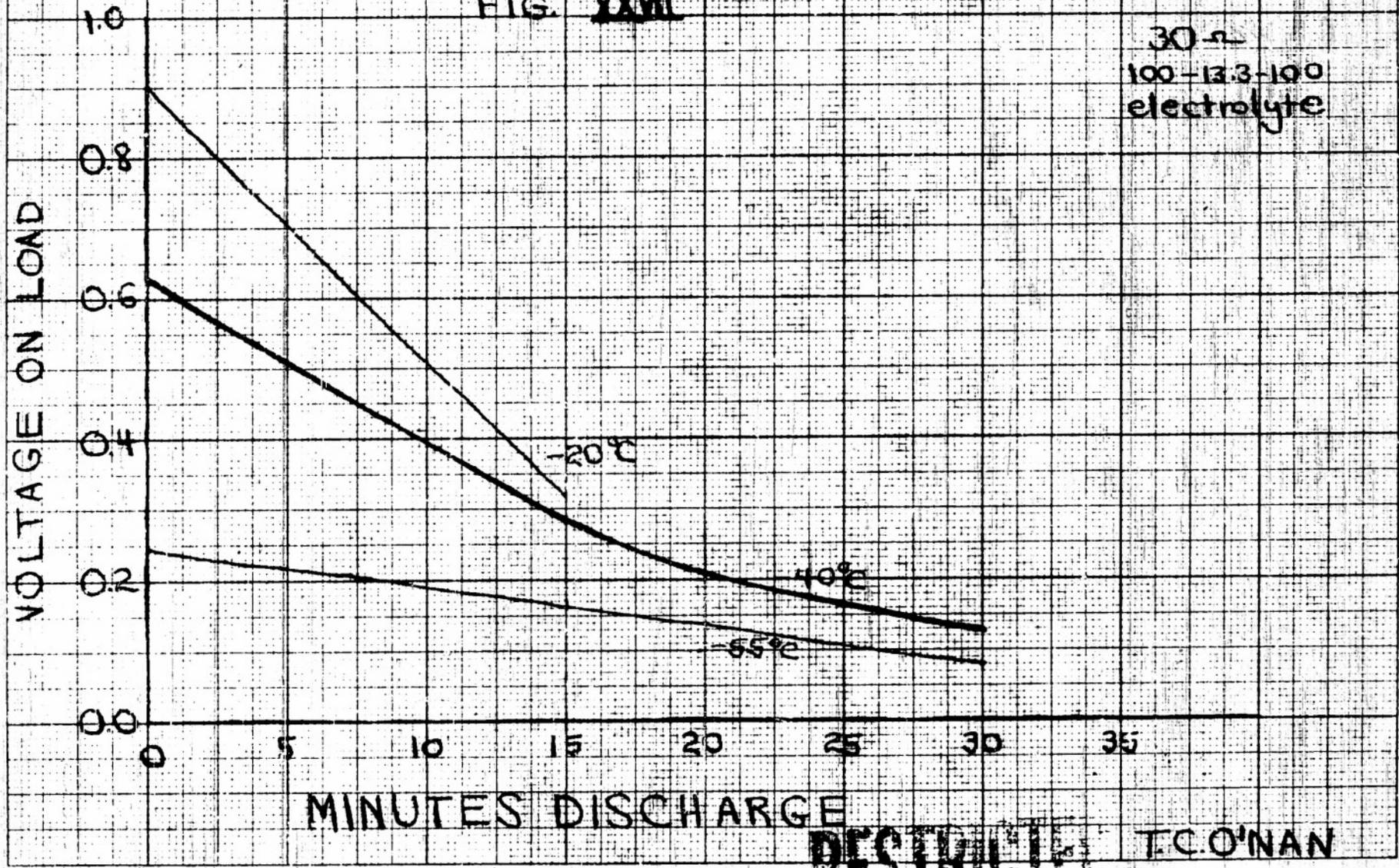
RMB-4 CELLS
LOW TEMPERATURE PERFORMANCE
FIG. XXVI



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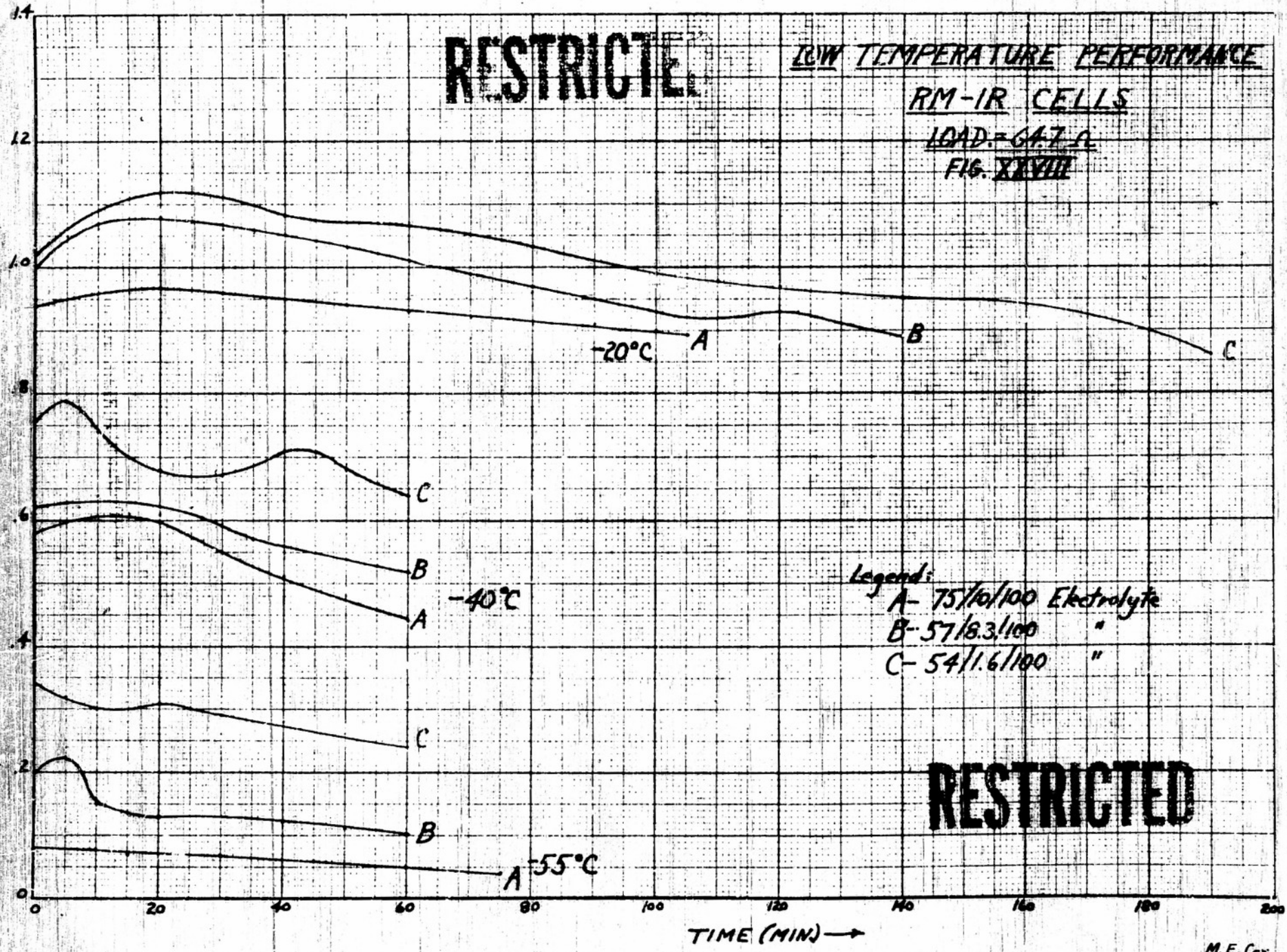

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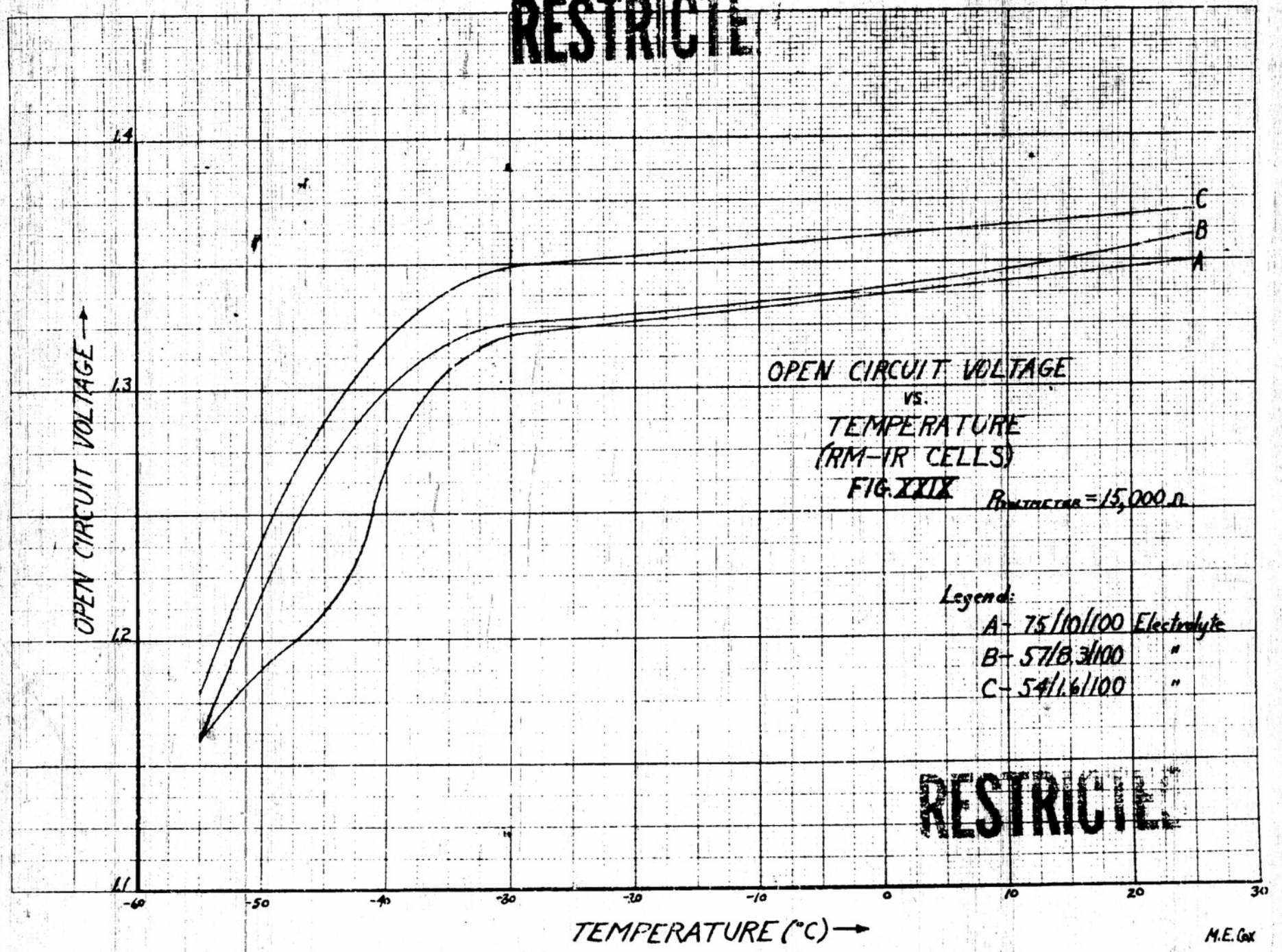

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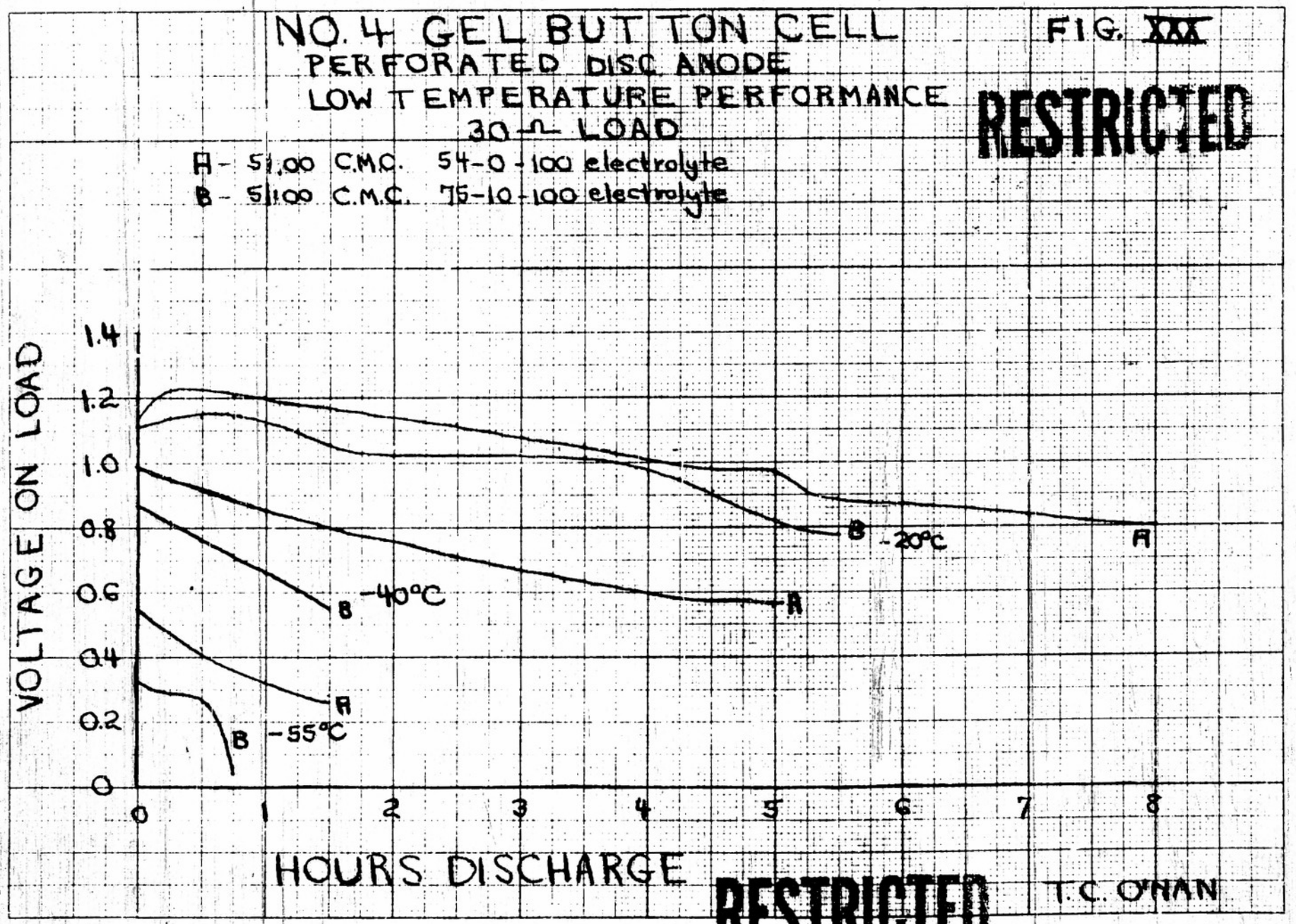
M.E. Cox



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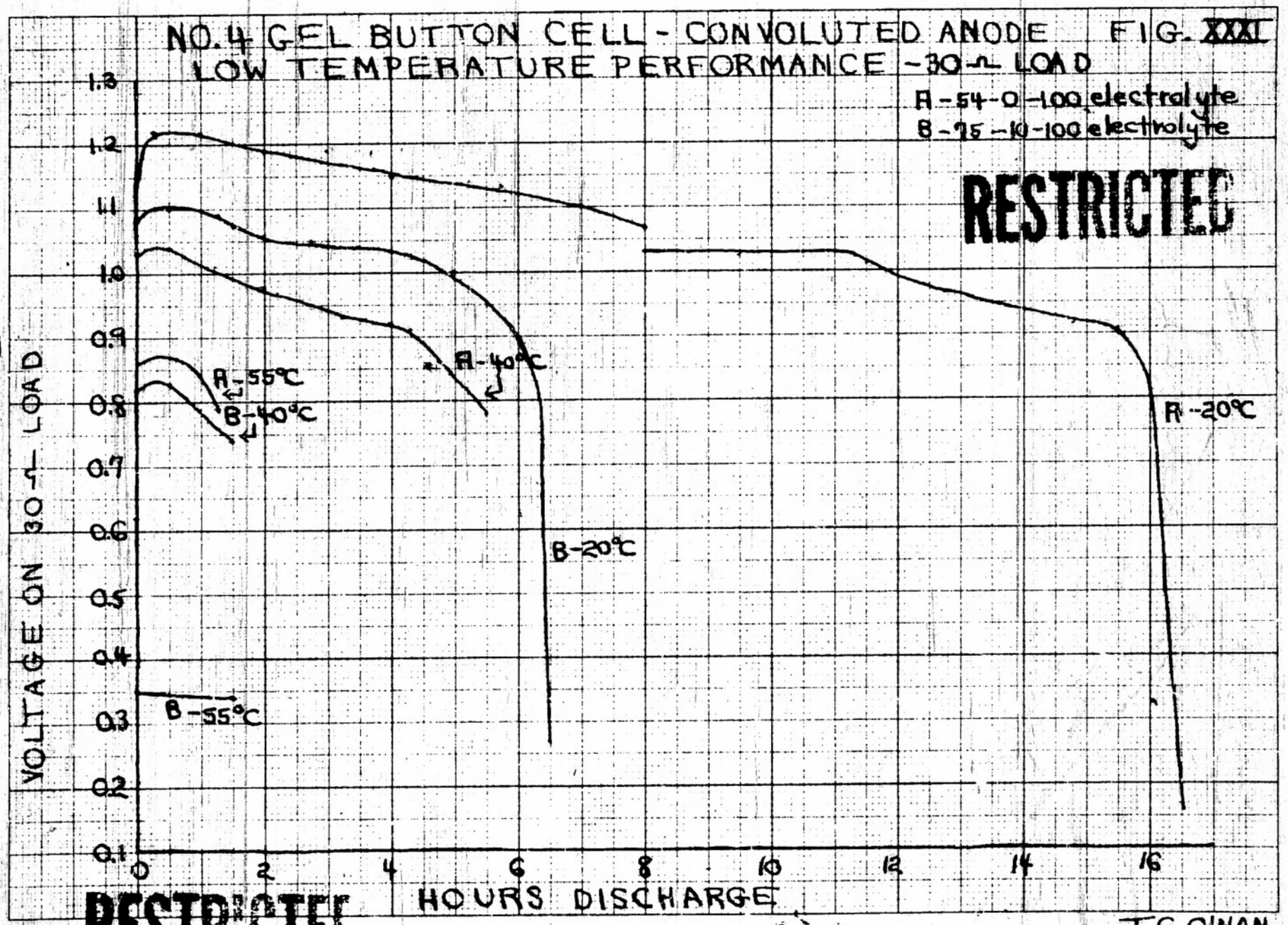

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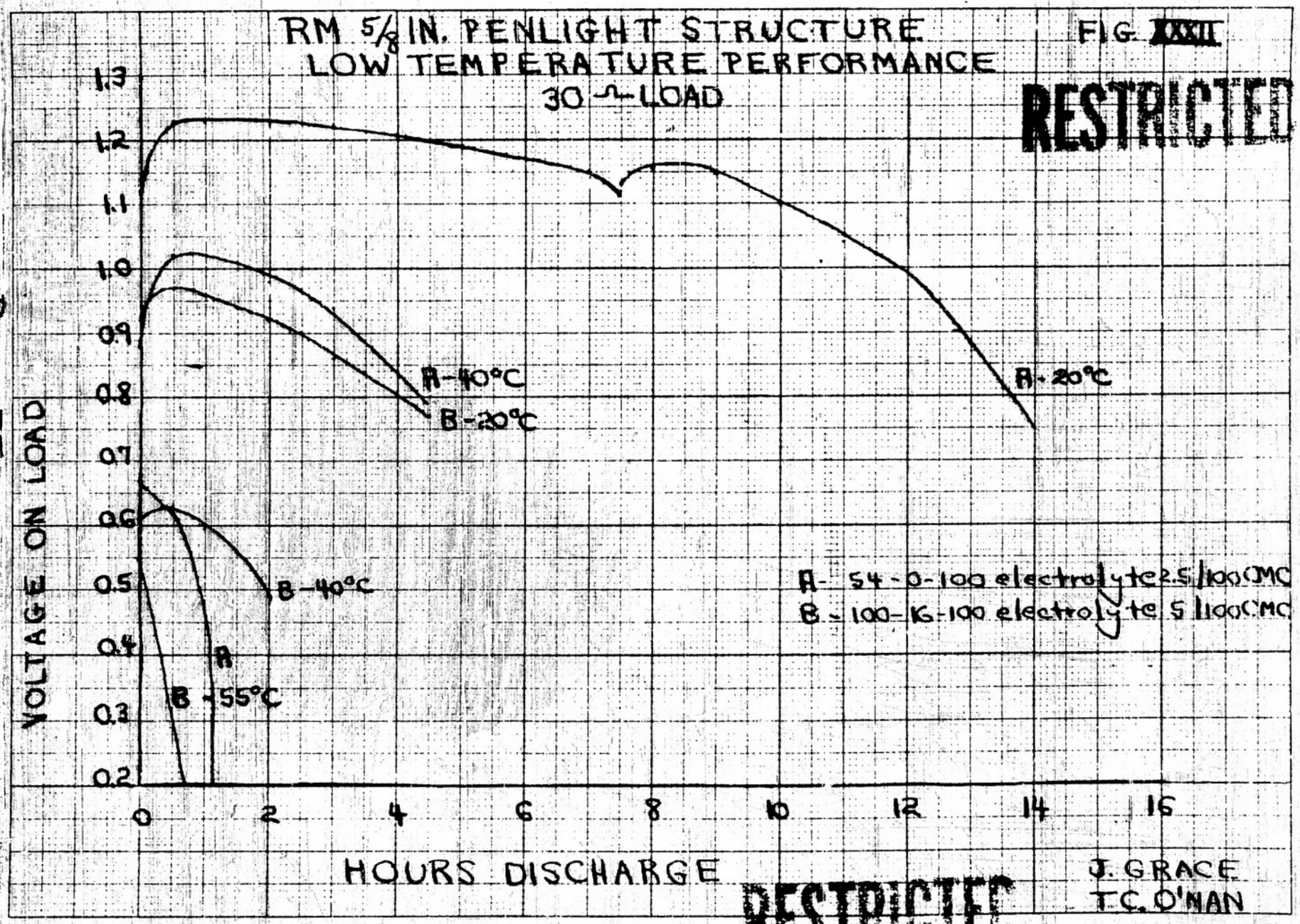

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