NASA CR-54295

INVESTIGATION OF

BATTERY ACTIVE NICKEL OXIDES

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
CONTRACT NO. NAS-34178

NASA LEWIS RESEARCH CENTER

SECOND QUARTERLY REPORT

September 12 to December 12, 1964

Prepared By:

P. Ritterman
Research Chemist

Approved By:

H. N. Seiger
Director of Research

GULTON INDUSTRIES, INC.
Alkaline Battery Division
Metuchen, New Jersey

TABLE OF CONTENTS

TABLE OF CONTENTS
(Continued)

TABLE OF CONTENTS
(Continued)

TABLE OF CONTENTS
(Continued)

PAGE NO.

TABLES & FIGURES

TABLE OF CONTENTS
(Continued)

TABLE OF CONTENTS
(Continued)

ABSTRACT

14211

The study of the effect of stand at various temperatures on the sintered plate and graphite nickel oxide electrodes was continued. Results indicate that the two types of positive electrodes differ considerably with respect to electrochemical and chemical characteristics.

The investigation into the effect of overcharge on the composition of the sintered plate and nickel oxide electrode has been initiated. The x-ray diffraction patterns of the positive electrode of a two plate Ni-Cd cell, under conditions of dynamic overcharge have many lines which cannot be attributed to NiOOH, $Ni(OH)_2$ or $Ni_2O_3 \cdot H_2O$.

The study concerned with the effect of state-of-charge on composition of sintered plate nickel oxide electrodes, at the C and C/10 rate, has been started.

Author →

I. INTRODUCTION

A. PURPOSE

Work performed under this contract is directed (1) toward an identification of the nickel oxide compounds formed during charge and discharge of the electrodes; (2) to determine when they are formed; (3) to determine which factors (such as current density and temperature) affect the charge process; (4) to relate the shelf loss process to these compounds, and (5) to determine the compounds which disappear during discharge of these electrodes.

In order to accomplish this a three phase program has been devised.

Phase I is directed toward a study of shelf life changes in the nickel electrode at room temperature and certain low and elevated temperatures.

Phase II deals with the forms of nickel oxides appearing at various states of charge, and also with the appearance of unstable oxides during dynamic overcharge.

Phase III deals with methods to prepare positive sintered plate electrodes which are stable at high temperatures with respect to shelf and cycle life.

II. OUTLINE OF PROGRAM

A brief review of the program, discussed in detail in the First Quarterly Report, is outlined below:

A. PHASE I

1. Literature Survey

An enumeration and summation of recent papers dealing with the determination of structure and composition of nickel oxide electrodes appeared in the First Quarterly Report.

2. Construction of Test Cells

Sintered and tubular type plates will be prepared entirely from the same batch of nickel nitrate solution. These plates will be used in construction of cells with excess negative capacity, distributed in such a manner as to completely surround the surface area of the positive electrodes.

3. Treatment

Thirty cores of each type will be constructed using 34% KOH and given five formation cycles. From the fifth discharge of these formation cycles, 21 cells closest in capacity will be chosen. These will then be charged at 25°C at C/10 to 200% of capacity. Cells will be grouped and allowed to stand at 25°C, 10°C and 50°C. After at least a 24 hour stand at these temperatures, cells will be analyzed and allowed to stand at their respective temperatures for a period of 3 months and then analyzed again.

4. Method of Analysis

The positive plates will be analyzed by chemical means, x-ray diffraction, differential thermal analysis, and spectrographically.

B. PHASE II

1. Unstable Oxides

It is during overcharge that unstable nickel oxides are formed. These unstable oxides of nickel, having short half-lives, must be analyzed during dynamic overcharge or immediately following the overcharge. For this purpose, a 2 plate cell will be prepared and assembled within a polyethylene bag. This cell will be designed so that it can be x-rayed while on charge and overcharge. In this manner, the appearance of strange lines can be followed.

2. Stable Oxides

The same type of cores used in Phase I will again be employed. Thirty-six of each type will be made. Charges and discharges will be run at the C and C/10 rates. Cores will be subjected to charges and discharges, resulting in cells whose positives will then be at various states of charge. After a 24 hour stand, these will be analyzed.

C. PHASE III

1. Literature Search

A survey will be made to determine whether oxides or hydroxides similar to those found in the nickel electrode have been stabilized for higher temperature tolerance.

2. Stable Oxide Synthesis, Test and Analysis

The nickel oxides will be prepared and tested by immersion in alkaline battery electrolyte at 40°, 50°, and 60° centigrade for a length of time deemed necessary for the perception of change in composition.

3. Electrode Preparation and Cell Fabrication

Methods found successful for stable oxide synthesis will be used to prepare sintered plate positive electrodes, which will subsequently be used in fabrication of nickel cadmium cells.

4. Stabilized Oxide Cell Testing

Two cells for each stabilization method, shall be cycled at 50°C. Two other cells shall be cycled at 70°C. After testing at these temperatures, the cells shall be cycled again at 25°C. Then they shall be charged fully and stored for a suitable period of time at 50°C to ascertain change in rate at which capacity is lost.

III. EXPERIMENTAL PROCEDURES & RESULTS

A. EXPERIMENTAL PROCEDURES

1. Phase I

(a) Preparation of Plates

The preparation of both sintered plates and tubular positives was discussed in detail in the First Quarterly Report.

(b) Construction of Cells

i. Sintered Plate Cells

The construction of these was completely described in the report for the previous quarter.

ii. Tubular Plates

The stainless steel tubes were filled with a mix of graphite and powdered $Ni(OH)_2$, prepared as described in the First Quarterly Report. The tube was sealed on the bottom, placed in a special jig, (Fig. 1) and then filled by tamping small amounts of active material at a time. After filling, a tab was welded to the top. Thus, the completed tubular positive electrode had the dimensions 4" x 7/32" I. D. Thirty such positive plates were constructed and surrounded with an excess of antipodic capacity in the form of sintered plate cadmium electrodes.

(c) Formation Cycling

i. Sintered Plate Cells

The treatment of these cells involved charge, discharge and shorting. The procedure was described in full detail in the previous quarterly report.

ii. Tubular Plates

Preliminary tests showed that current densities corresponding to the C/5 rate, based on theoretical capacity could not be used due to the high voltage and immediate oxygen gassing at the positive. Edison's patent #678722, by which these plates were made, called for an extended charge at a current density of 50 ma/in^2. This corresponded to 155 milliamperes, which we found was also too great.

Empirically, it was found that a 50 ma charge gave satisfactory results. It was also empirically established that a discharge rate of 50 ma corresponded essentially to the C/3 discharge rate.

Based on these results, the formations were performed. Each cell was given 5 cycles, involving a 50 ma charge for 16 hours followed by individual discharges through a 22 ohm resistor. This was followed by a 24 hour short. During the first three cycles, the cells were discharged to 0.2V before shorting. The last two discharges were taken to 1.0V.

Based on the results of the 5th discharge, 21 cells closest in capacity were chosen out of the group of 30. These, in turn, were divided into three groups of 7 cells each, according to capacity; high, medium, and low. This procedure is identical with that previously used for the sintered plate cells.

(d) Preparatory Self Discharge Measurements and Analysis

i. Sintered Plate Cells

The procedure followed was described in our previous report. It went above and beyond the original work plan, in that both wet and dry type positive plates were analyzed. For details, see pages 15 and 16 of Reference 1.

At the end of this quarter, the sintered plate cells had completed their 3 months stand at 25°, 10° and 50° centigrade. From the analysis of previous x-ray data obtained during the first quarter, and as was stated in the First Quarterly Report, all x-ray work done during this quarter was with the positive plates in the "wet" condition. It was also found that little difference existed among the high, low, and medium capacity groups with respect to x-ray characteristics.

Therefore, only 4 positive plates were analyzed via x-ray diffraction; one which had stood 3 months at a temperature of 25°C, one which had the same stand time at a temperature of 10°C, and two that stood for that duration at 50°C. Three plates from the low capacity group were used for this, and, as a check, an additional plate for the higher capacity group was used at 50°C.

ii. Tubular Plate Cells

Based on statistical distribution of capacity for the 21 cells, it was decided that six cells would be sufficient for this test. This was supported by the fact that in previous tests run on sintered plates, there was little difference among x-ray characteristics of the high, low, and medium group. The six cells selected had 2 representatives of each group based on the

discharge of the fifth cycle. The 6 cells were placed in an oil bath, kept at 25°C, and there charged at 0.015 amperes for a period of 20 hours.

One cell was allowed to stand on open circuit in the oil bath for a period of 24 hours. At this point, the cell was taken apart, the steel tube opened, its active material placed in a sample holder, and immediately x-rayed. Another cell was removed from the oil bath after being charged and allowed to stand 24 hours at 10°C. A third was allowed to stand for 24 hours at 50°C. The latter two cells were opened, active material removed and x-rayed as described above.

The three remaining cells will be allowed to stand for 3 months, with one cell kept at each of the above mentioned temperatures.

2. Phase II

(a) Unstable Nickel Oxides

A two plate cell having a positive electrode of the dimensions 1 x 2 x 0.09 cm., and a negative electrode of the same thickness and twice the area, was contained within a polyethylene bag. The positives and negatives had tabs, extending through the bag, enabling the cell to be charged.

The cell was charged for 1 hour at the C rate (50 milliamperes) based on positive capacity. It was then placed in the goniometer of the x-ray apparatus. The charge was continued at 50 ma while the cell was being x-rayed. A sweep between the angles $2\theta = 10^{\circ}$ and 80° was made, and after its completion, the x-ray apparatus was

returned to 10^{o} as the charge continued. This was done for a total of 6 sweeps, corresponding to an overcharge of 350% at the C rate. After the 6 sweeps, the cell was placed on open circuit and the x-raying continued for 3 additional sweeps between these angles. This corresponds to the cell being on open circuit from 0 to 105 minutes.

The x-ray apparatus was then shut off, and after 20 hours on open circuit, the cell was x-rayed again between the angles of $2\theta = 10^{o}$ and 80^{o} twice, to show duplication.

During overcharge, many strange lines were found between $2\theta = 55^{o}$ and 65^{o}. A discharged cell (duplicate to cell described above) was first x-rayed on open circuit and then x-rayed twice while on continuous charge at 10 ma.

The charge was continued with the current raised to 50 ma, during which time the cell was twice x-rayed between the angles of $2\theta = 55^{o}$ and 65^{o}.

(b) Stable Nickel Oxides

Thirty-six sintered plate cells, identical to those used in Phase I, were employed here. Although not previously planned, it was deemed advisable that, before any testing with respect to the electrochemical and chemical characteristics was begun, these cells be given formation cycles. The formation consisted of 5 cycles involving charge, discharge through a constant load (2.4 ohms) to 1.0V, followed by a dead short.

3. Special Tests

(a) Determination of Effect on Positive Plate Characteristics With Respect to Sealed and Vented Charge.

A test to show a similarity of compounds in the positive of a positive limiting cell can be based on the number of coulombs passed through anodes during charge before a rapid rise in pressure is noted.

A sealed electrolyte starved VO-6HS cell equipped with a pressure gauge and evacuated, was charged and discharged for 4 cycles in the following manner; (1) charged at 1.2 amperes until a rapid rise of pressure, with respect to time, was noted. The charge was then continued for a short time beyond this point. (2) Discharged at 3.0 amperes to 1.0V. (3) Dead shorted overnight.

After completion of the 4 cycles, the cell was flooded with KOH, vented to the atmosphere, and then resealed. Five additional cycles were now given to this cell. Each cycle consisted of; (1) 1.2 amp charge for a time slightly beyond a rapid pressure rise (2) a discharge to 1.0V at 3 amps. (3) an overnight short. (4) venting to atmospheric pressure and resealing.

(b) Analysis of M-2 Graphite Flake

The graphite flake used as a conducting material in the tubular positive plates was quantitatively analyzed for impurities. This was done by means of spectrographic analysis at Ledoux & Co. in Teaneck, N. J.

(c) Extended Charge of Freshly Prepared Tubular Plate

A freshly prepared tubular plate, never having received or given forth coulombic energy, was made into a cell by surrounding it with excess antipodic material. This cell was continuously charged for 100 hours at 50 milliamperes. On subsequent discharge, the cell showed unusually high capacity. It was charged again, this time at 120 ma for a period of 16 hours. After a partial discharge at 50 ma, the cell was given a continuous charge at various rates and continued until vigorous gassing was noted, off the positive electrode. When vigorous gassing was noted at 100 ma, the charge was discontinued and after an overnight stand, the active material of the positive plate was x-rayed.

B. DATA

1. Phase I

(a) Sintered Plate Cells

The listing of the various d spacings obtained from the 18 x-rays of sintered plate positives appear in Table I.

The 4 positive plates which were studied after a 3 month stand at the previously mentioned isothermal conditions, came from cell numbers 16, 11, 14 and 17. These correspond to stands at temperatures of 10°C, 25°C, 50°C, 50°C respectively. The x-ray diffraction patterns obtained from the positives appear again respectively in Figures 2, 3, 4 and 5.

(b) Tubular Plate Cells

i. Formation

The capacities of 30 tubular plate cells expressed in minutes to 1.0V appear in Table II. The discharges through a

22 ohm resistor, for the 1st and 5th cycles, are given. The letters H, M, L, next to cells in that table, indicate that these were selected as the 21 closest in capacity, and also to which group they were relegated using the letters for high, medium and low capacity, based on the discharge to 1.0V during the 5th cycle.

Figure 6 shows a typical discharge curve through the 22 ohm resistor when discharge was allowed to proceed beyond the 1.0V cut-off, as was done in the first 3 discharges.

ii. Preparatory Self-Discharge Measurements and Analysis.

The six cells chosen for this test were numbers 2, 8, 10, 16, 24, and 30. All were treated as described in the previous section with respect to thermostated charge. After 24 hour stands on open circuit at temperatures of 25°C, 10°C and 50°C, x-ray diffraction patterns were obtained from active material in the positives of cell numbers 10, 30, and 16 respectively. These appear in the same sequence in Figures 7, 8 and 9. Figure 10 is an x-ray diffraction of the active material mix to which no coulombic energy had been given or taken.

2. Phase II

(a) Unstable Nickel Oxides

The experimental set-up shown in Figure 11, utilizing the previously described two plate cell, was used for this test. The positive plate, naturally, was placed face up in the goniometer. X-ray diffraction patterns were taken after the cell had received a full charge with respect to the positive. Figures 12, 13, and 14 are the patterns obtained during the first 3 sweeps between 2θ = 10° and 80°. This corresponds to an overcharge time of 105 minutes,

which, at the C rate of charge, is 175% overcharge.

Figures 15, 16, and 17 are all patterns as the sweep between the previously mentioned angles and charge at the C rate continues. At the completion of sweep, represented by Figure 17, our cell has been on continuous overcharge for 350% of its rated capacity at the C rate.

At this point, the charging current was turned off, thus placing the cell on open circuit, but the x-ray sweep continued thrice more, as shown in Figures 18, 19, and 20. At the end of the sweep shown in Figure 20, the cell had completed an open circuit stand of 105 minutes.

After a 20 hour additional stand, the cell was again x-rayed between $2\theta = 10^{\circ}$ and 80° twice, to show duplication and stabilization. See Figures 21 and 22.

To further check on the results, a discharged cell, duplicate to the one used above, was first x-rayed on open circuit, then twice while on charge at 10 ma between the angles of $2\theta = 55^{\circ}$ and 65°. The charging current was increased to 50 ma as the sweep between the 10 degrees continued for two more times. The entire x-ray diffraction data appears in Figure 23.

(b) Stable Nickel Oxides

The discharge data for the 5 formation cycles of the 36 sintered plate cells appear in Table III. The cells were discharged individually through 2.4 ohm resistors to 1.0 V.

3. Special Tests

(a) Determination of Effect on Positive Plate Characteristics With Respect to Sealed and Vented Charge

The data obtained during charging of VO-6 H.S. cells were plotted for each cycle in the form of pressure vs. ampere hour or coulombic input. Figure 24 is a plot of results obtained from the fourth "sealed" charge. Figure 25 is a plot of pressure vs. coulombic input during flooded "vented charge" on the cycle immediately following the fourth and final "sealed" charge.

(b) Analysis of M-2 Graphite Flake

The impurities and their amounts, found in the M-2 Graphite Flake, used as a conducting material for the tubular plates, are shown in Table IV.

(c) Extended Charge of Freshly Prepared Tubular Plates

After an input of 5.00 ampere hours at 50 ma, a total of 0.427 ampere hours were obtained before the cell went below 1.0 V. The discharge was carried out, for the most part, at 50 ma. After 6 hours, the cell voltage was above 1.10 V, so current was gradually raised to 300 ma, at which point cell voltage began to decay. The discharge was returned to 100 ma, at which point the cell voltage dropped below 1.0 V after 10 minutes at this rate.

Immediately thereafter the cell was charged for a total of 1.9 ampere hours and then discharged at 50 ma for 7 hours. Cell voltage remained above 1.10V during that entire time.

After an overnight stand, the cell was charged for a total of 0.450 AH at various rates ranging from 100 to 400 ma. The cell received 15 minutes of charge at the latter rate before vigorous gassing of the positive plate was noted. After that point, an additional charge of 2½ hours at 100 ma was given to the cell before vigorous gassing of the positive was noted.

The x-ray diffraction patterns obtained from this plate, following overnight stand, are shown in Figure 26.

C. DISCUSSION

1. Phase I

(a) Sintered Plate Cells

The "d" spacings in Table I indicate that there exists little difference between positives of high, low and medium capacity, with respect to their crystal structures and composition. Differences do exist between the washed and dried plates and the undisturbed wet plates. This was found true at all three temperatures, though, strangely enough, a decrease in the number of "d" spacings results from the washing and drying of these plates. Also, as temperature of stand increases, the number of lines found increases correspondingly.

For the most part, we can say that the compounds formed at all temperatures, on the basis of the literature reports, were $Ni(OH)_2$ and β' NiOOH. At the time these x-ray patterns were obtained, our

techniques were not to be considered highly refined. It has appeared that this work is complex so that our techniques were since considerably refined.

These refined techniques were used in the recent data obtained under this contract when x-raying the plates which completed their three month's charge stand at temperatures of 10^{o}, 25^{o}, and $50^{o}C$. Although not yet thoroughly analyzed, these recently obtained data show an increase in peak heights of nickel oxide compounds, when compared to those found after 24 to 72 hour stands, as shown in the First Quarterly Report. Further, the previously intense peaks of sintered nickel have shown signs of waning. The higher the stand temperature, the greater is the increase in nickel oxide peaks and the greater, too, is the waning of the sintered nickel peaks. This is hypothesized to be due to a migration of active material, or crystal growth of active material which masks the previously intense nickel peaks. This can be seen by comparing the x-ray diffraction diagrams given in Figures 2, 3, 4 and 5 of this report with the appropriate figures of the First Quarterly Report.

(b) Tubular Plate Cells

The necessity for low rate charges, with respect to both surface area and theoretical capacity, indicates a major difference between tubular plates and sintered nickel plates. This behavior possibly explains why the original Edison process, using graphite had been abandoned in favor of a process employing flake nickel. The evolution of oxygen during charge at current densities recom-

mended by Edison for initial treatment results in a very ineffective charge.

The x-ray data obtained from tubular plate active material, after a 24 hour charged stand indicate a structure of essentially a mixture of $Ni(OH)_2$ and β NiOOH. This is true at all three temperatures. There exists, however, a considerable difference between the "cycled" charged active material and that unexposed to coulombic energy (compare Figures 7, 8, and 9 with 10). Due to inefficiency of charge and the discharge capacities found from these pocket plates, it is reasonable to assume that the active material is predominately $Ni(OH)_2$. The not-previously-charged material, shown in Figure 10, is pure $Ni(OH)_2$. The differences found between these two structures may be due to various phases of $Ni(OH)_2$ as suggested recently by Tuomi.

2. Phase II

(a) Unstable Nickel Oxides

Although just recently performed, a cursory examination of the x-ray data taken on overcharged sintered plates shows the existence of strange lines which arise during overcharge, remain as long as overcharge is continued, and wane during open circuit. The fact that no such peaks were found between angles of $2\theta = 55^{\circ}$ to 65° when the cell was being charged, proves that these peaks do not arise because of the passage of coulombic energy. Though there is even more to be inferred from the data, this shall be delayed until the experiments are completed, during the next quarter, and interpreted.

(b) Stable Nickel Oxides

The data in Table III need no discussion. These are the capacities of the cells prepared for Phase II.

3. Special Tests

(a) Sealed versus Vented Cell Charges

Sealed cells generally are semidry, while vented cells contain mobile electrolyte. The question of study of open cells was raised, and this experiment was devised to provide insight into the answer. The coulombic efficiency of a semidry cell was compared to a vented-flooded cell. Reasoning that similar coulombic inputs would result in the formation of the same compounds or species, a decision could be made based upon the experimental results.

The experimental results are shown in Figures 24 and 25. Because the rise in pressure occurs at nearly the same time for the same cell in the two different conditions, it is concluded that the coulombic efficiencies are also nearly the same. From this, then, is the justification of carrying out the experiments in the semidry but unsealed manner.

(b) Analysis of M-2 Graphite Flake

The results of the analysis of the graphite flake are self-explanatory. Several elements (Al, Fe, Si) are present in relatively large quantities. No action is contemplated because of the behavior of the tubular plates, which is discussed in the next section.

(c) Extended Charge of Freshly Prepared Tubular Plate

The results of the test using extended overcharge of "not previously used" tubular plates at low rates, indicate that this kind of formation may be necessary before these plates can be subjected to higher current densities, the higher current densities being comparable to those used in the sintered plate. The x-ray diffraction patterns of these tubular plates indicated peaks which are characteristic of γ NiOOH, or, using Tuomi's terminology, α nickelate. These findings on the tubular plates raises a question on the value of continuing work with them. Because α nickelate is found only in the high rate formation charge to the extent of 10%, analysis accuracy is necessarily decreased by an order of magnitude. Tubular plates formed by extensive overcharge at low current density are of no value in this program, because compounds not similar to those on the sintered plate are found.

D. CONCLUSIONS

1. During overcharge of sintered plate nickel oxide electrodes, compounds with relatively short half lives are formed. The are present in addition to β NiOOH and $Ni(OH)_2$.

2. Stable compounds other than β NiOOH and $Ni(OH)_2$ are, under certain conditions of overcharge, found in graphite tubular type electrodes.

3. During extended stand, especially at high temperatures, there is an indication of a crystal growth, and also a masking of sintered nickel surface. This is tentatively ascribed to a migration of active material from the pores toward the surface of the electrode.

4. The electrochemical and chemical properties of the tubular positive electrodes are dependent on their histories or previous usage.

IV. WORK FOR THE NEXT QUARTER

A. COMPLETION OF OVERCHARGE EXPERIMENT

The appearance and disappearance of unstable nickel oxides, with respect to their individual peaks, will be studied. The rate at which this occurs will also be investigated.

B. ANALYSIS OF POSITIVE PLATES

1. X-ray

X-rays obtained from sintered plate positives during dynamic overcharge will be analyzed.

2. Chemical analysis

A group of sintered plates will be prepared and analyzed chemically. An attempt will be made to separate the elemental Ni magnetically. A compositional analysis will be used to determine formula weight, and the equivalent oxidation state.

3. D. T. A.

The study of both pocket and sintered plates will be performed during the next quarter under inert, oxidizing and reducing atmospheres.

C. PHASE II - STABLE OXIDES - EFFECT OF STATE OF CHARGE

It is expected that we will complete this work, with respect to the sintered plate electrodes, during the next quarter.

D. "GRAPHITIC" LEVEL

The fact that both sintered and tubular plate positive electrodes exhibited a "graphite level" plateau at around 0.7V is of interest. We plan to discharge both types of electrodes to that point, and then analyze their structure by the various means available to us.

V. REFERENCES

1. Investigation of Battery Active Nickel Oxides -
First Quarterly Report - P. Ritterman and H. Seiger

2. T. A. Edison - Reversible Galvanic Battery
Patent #678,722.

3. D. Toumi - The Forming Process in Ni Positive Electrodes,
Journal of the Electrochemical Society
January 1965.

TABLE I

LIST OF "d" SPACINGS

FIGURES 2-19 OF FIRST QUARTERLY REPORT

FIGURE NO.	TREATMENT OF PLATE: TEMP.	STANDING TIME BEFORE X-RAY	CONDITION OF PLATE	CELL NO.	"d" SPACINGS OTHER THAN OBTAINED FROM NICKEL							
2	25°C	24 HOURS	WET-UNWASHED	21	4.68	3.75	2.97.		2.44	2.34		
4	25°C	48 HOURS	"	"	5	4.68	3.76	—		2.47	2.36	
6	25°C	72 HOURS	"	"	20	4.61	3.73	2.91		2.49	2.34	
3	25°C	24 HOURS	WASHED-DRY		21	4.68					2.34	
5	25°C	48 HOURS	"	"	5	4.65					2.32	
7	25°C	72 HOURS	"	"	20	4.63					2.34	
8	50°C	24 HOURS	WET UNWASHED		28	4.73	373		2.73	2.47	2.35	1.57 1.48
10	50°C	48 HOURS	"	"	15	4.56	3.69		2.71	2.48	2.34	1.57 1.48
12	50°C	72 HOURS	"	"	27	4.54	3.77	3.02 287	2.77	2.46	2.36	1.57 1.48
9	50°C	24 HOURS	WASHED-DRY		28	4.66						1.56 1.48
11	50°C	48 HOURS	"	"	15	4.65						1.56 1.48
13	50°C	72 HOURS	"	"	27	4.63						1.56 1.48
14	10°C	24 HOURS	WET-UNWASHED		24	4.63				2.47	2.36	
16	10°C	48 HOURS	"	"	10	4.54				2.47	2.36	
18	10°C	72 HOURS	"	"	25	—				2.47	—	
15	10°C	24 HOURS	WASHED-DRY		24	4.61					2.32	— —
17	10°C	48 HOURS	"	"	10	4.63					2.30	1.56 1.48
19	10°C	72 HOURS	"	"	25	4.59					2.30	1.53 1.48

TABLE II

CAPACITIES OF TUBULAR CELLS DISCHARGED THROUGH 22 OHM RESISTOR

CELL #	CYCLE #1 Minutes to 1.0 V	CYCLE #5 Minutes to 1.0 V	
1	146	168	M
2	145	193	H
3	157	191	H
4	168	192	H
5	162	200	
6	96	105	
7	190	205	
8	177	175	M
9	169	187	M
10	175	173	M
11	184	256	
12	172	193	H
13	182	195	H
14	160	172	M
15	170	166	M
16	191	190	H
17	127	127	
18	100	103	
19	133	115	
20	116	130	
21	132	162	L

TABLE II -- CONTINUED

CELL #	CYCLE #1 Minutes to 1.0 V	CYCLE #5 Minutes to 1.0 V	
22	137	154	L
23	159	169	M
24	150	160	L
25	125	145	L
26	106	75	
27	131	133	L
28	167	190	H
29	122	130	L
30	144	162	L

TABLE III

PHASE II - CAPACITIES OF SINTERED PLATE CELLS WHEN DISCHARGED THROUGH 2.4 OHM RESISTOR

CELL NUMBER	CYCLE #1	CYCLE #2	CYCLE #3	CYCLE #4	CYCLE #5
	Minutes to 1.0 V				
1	188	184	169	164	172
2	191	193	182	174	180
3	163	157	151	150	148
4	166	165	147	155	159
5	182	186	167	187	179
6	190	198	177	167	178
7	169	165	149	153	163
8	180	174	166	162	168
9	174	179	171	171	174
10	184	177	169	177	173
11	234	190	174	206	185
12	197	179	166	164	192
13	176	185	163	203	166
14	195	199	176	176	167
15	193	193	173	168	152
16	197	190	175	201	166
17	160	173	156	161	163
18	190	197	172	174	165
19	185	192	170	172	176
20	186	198	171	210	169
21	181	238	167	188	156
22	193	195	170	196	158

TABLE III -- CONTINUED

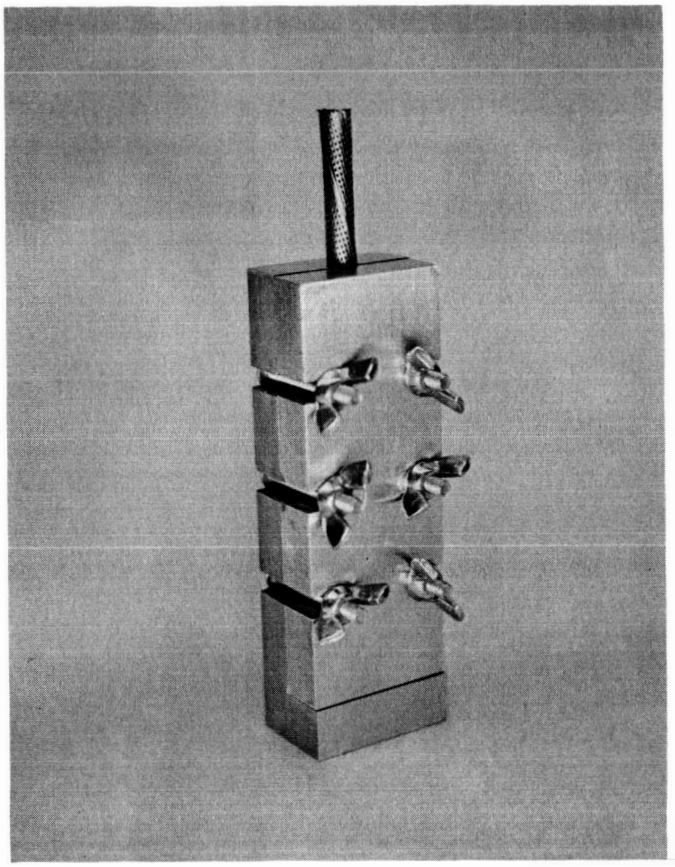
CELL NUMBER	CYCLE #1	CYCLE #2	CYCLE #3	CYCLE #4	CYCLE #5
	Minutes to 1.0 V				
23	208	193	177	186	180
24	186	207	173	173	177
25	196	182	167	165	177
26	196	189	171	175	161
27	172	185	162	161	168
28	173	173	163	197	177
29	199	192	173	174	169
30	184	181	166	161	152
31	183	179	172	154	153
32	177	171	163	159	180
33	128	161	150	155	148
34	128	182	164	163	154
35	195	192	169	172	177
36	188	191	173	190	157

TABLE IV

QUANTATIVE ANALYSIS VIA SPECTROGRAPH OF M-2 GRAPHITE FLAKE

IMPURITY	RELATIVE AMOUNT
Aluminum	0.5 %
Barium	0.002%
Calcium	0.02 %
Cobalt	0.001%
Copper	0.002%
Iron	0.5 %
Magnesium	0.02 %
Manganese	0.01 %
Molybdenum	0.002%
Silicon	1% - 10%
Titanium	0.005%
Vanadium	0.005%

FIG. 1



JIG *for* HOLDING TUBULAR PLATES DURING FILLING

GULTON INDUSTRIES INC.
METUCHEN, N. J.

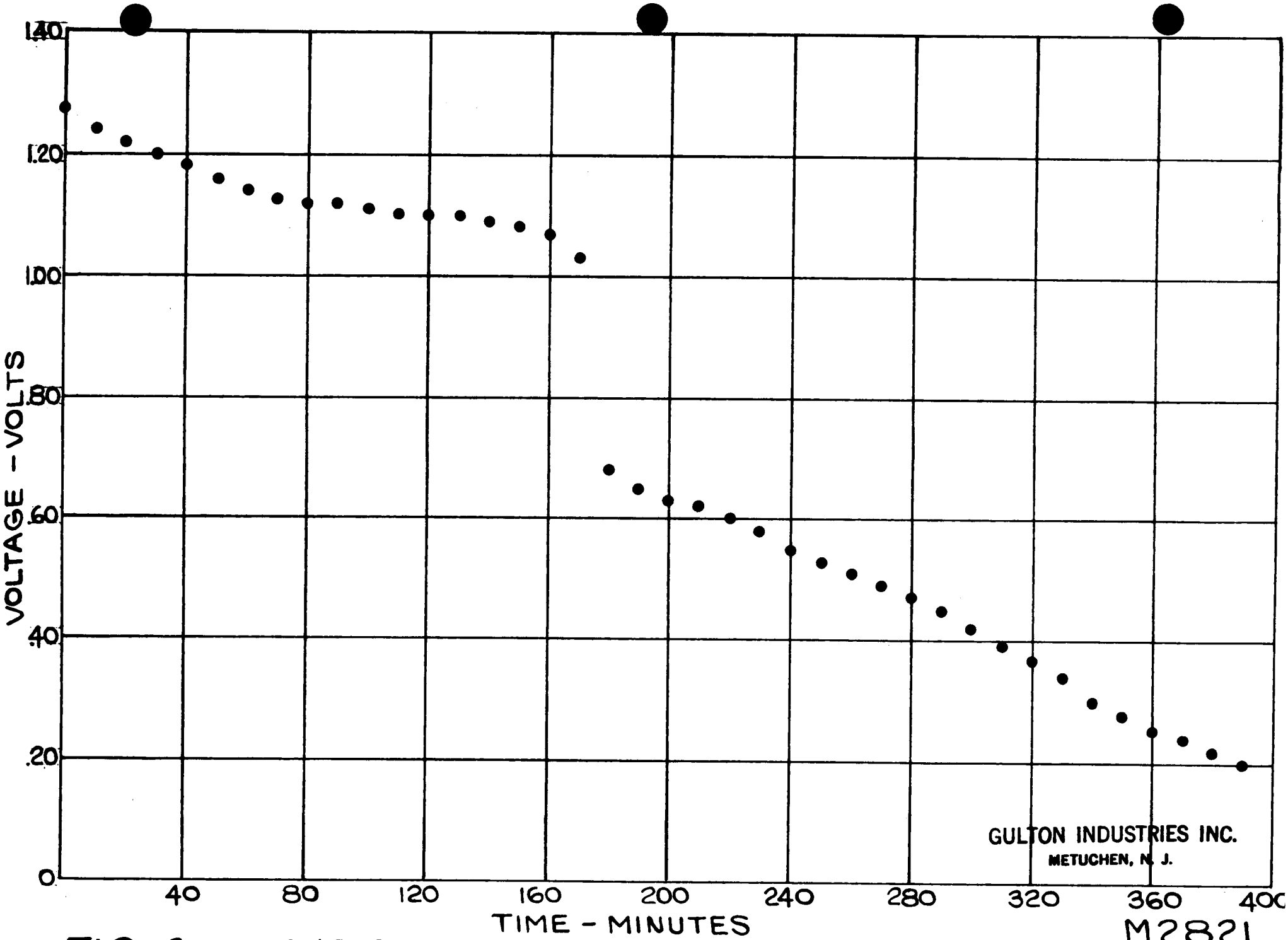
M2819

FIG 2

FIG. 6

KEUFFEL & ESSER CO.
MADE IN U.S.A.
100
90
80
70
60
50
40
30
20
10
0

FIG 5
BRUNSWICK
BRUNSWICK



M2821

FIG. 6 TYPICAL DISCHARGE CURVE TUBULAR PLATE CELL DISCHARGED THRU 22 Ω RESISTOR TO 0.2 VOLTS

FIG 1

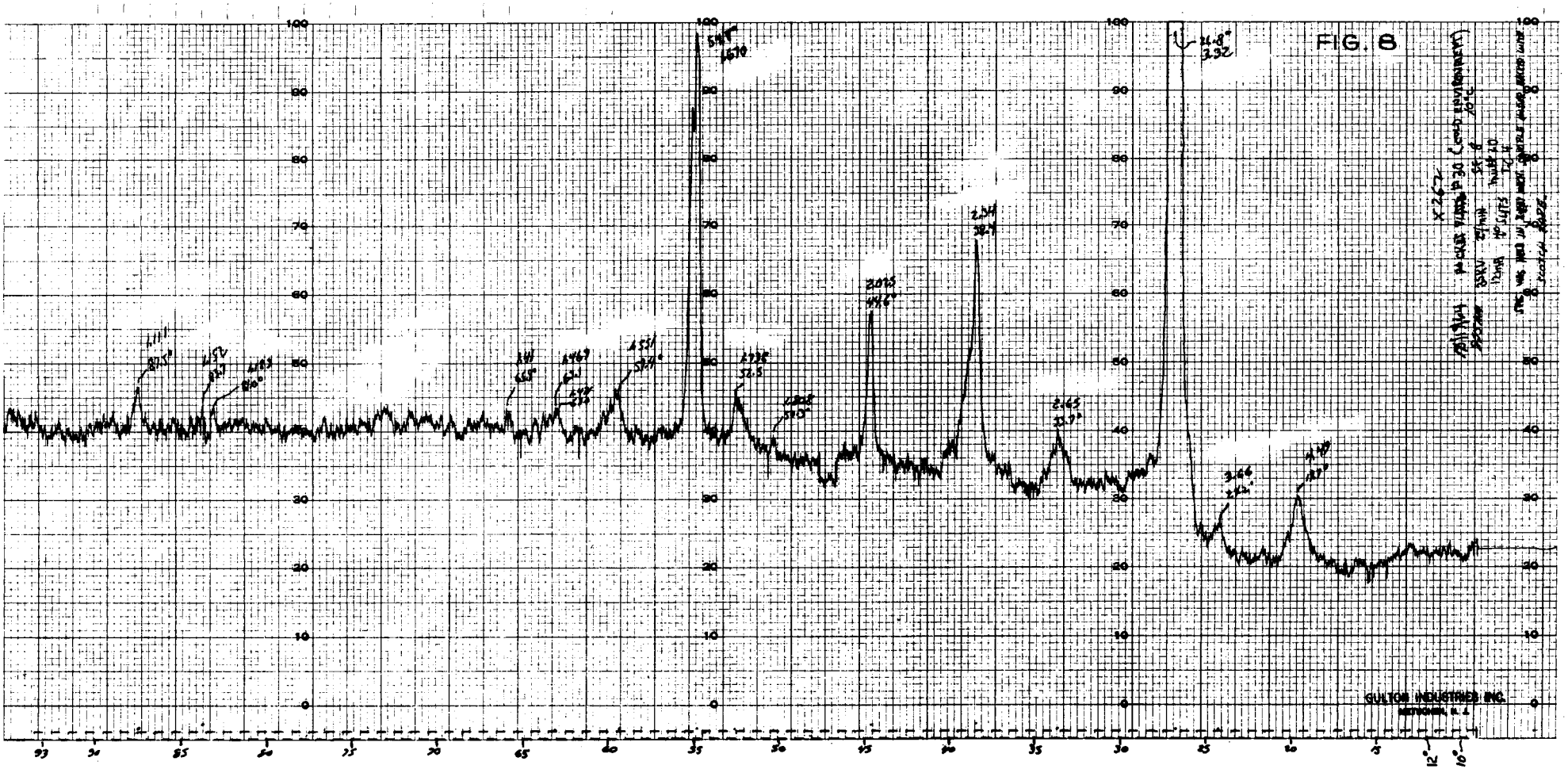


FIG. 8
GULTON INDUSTRIES INC.

FIG. 9

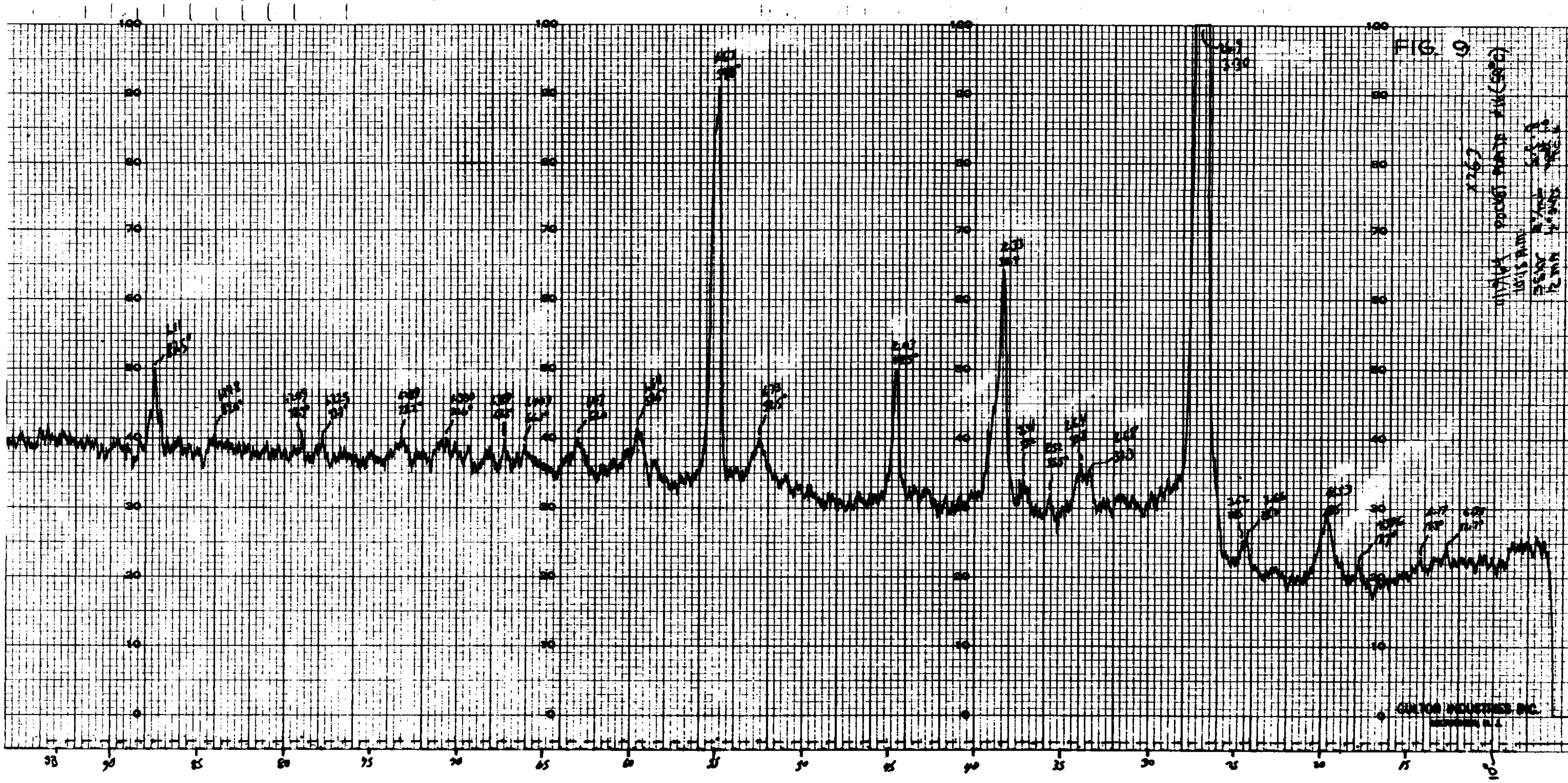
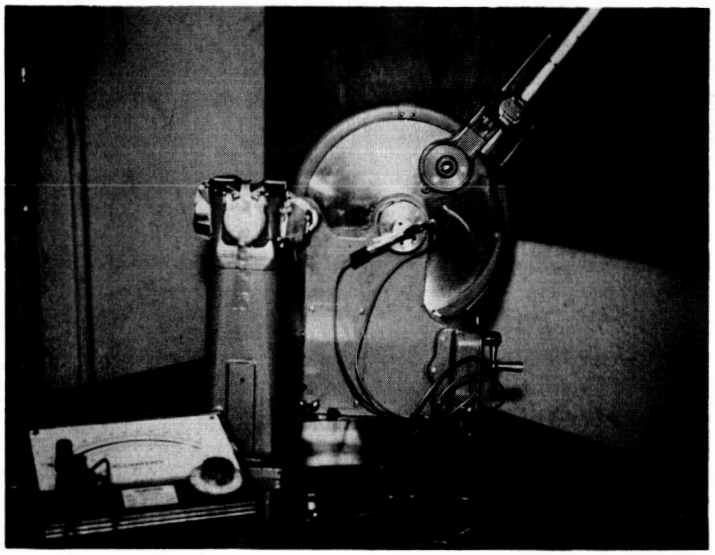


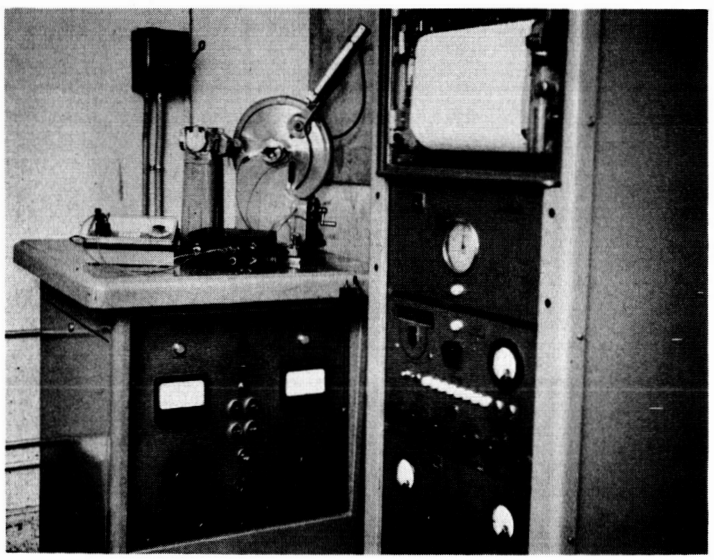
FIG. 10

FIG. 11

UNSTABLE NICKEL OXIDE EXPERIMENTAL SETUP



CLOSE-UP SHOWING CHARGER, METER, CELL AND GONIOMETER



LONG SHOT SHOWING X-RAY APPARATUS
RECORDER, CHARGER, METER, AND CELL

GULTON INDUSTRIES INC.
METUCHEN, N. J.

M2804

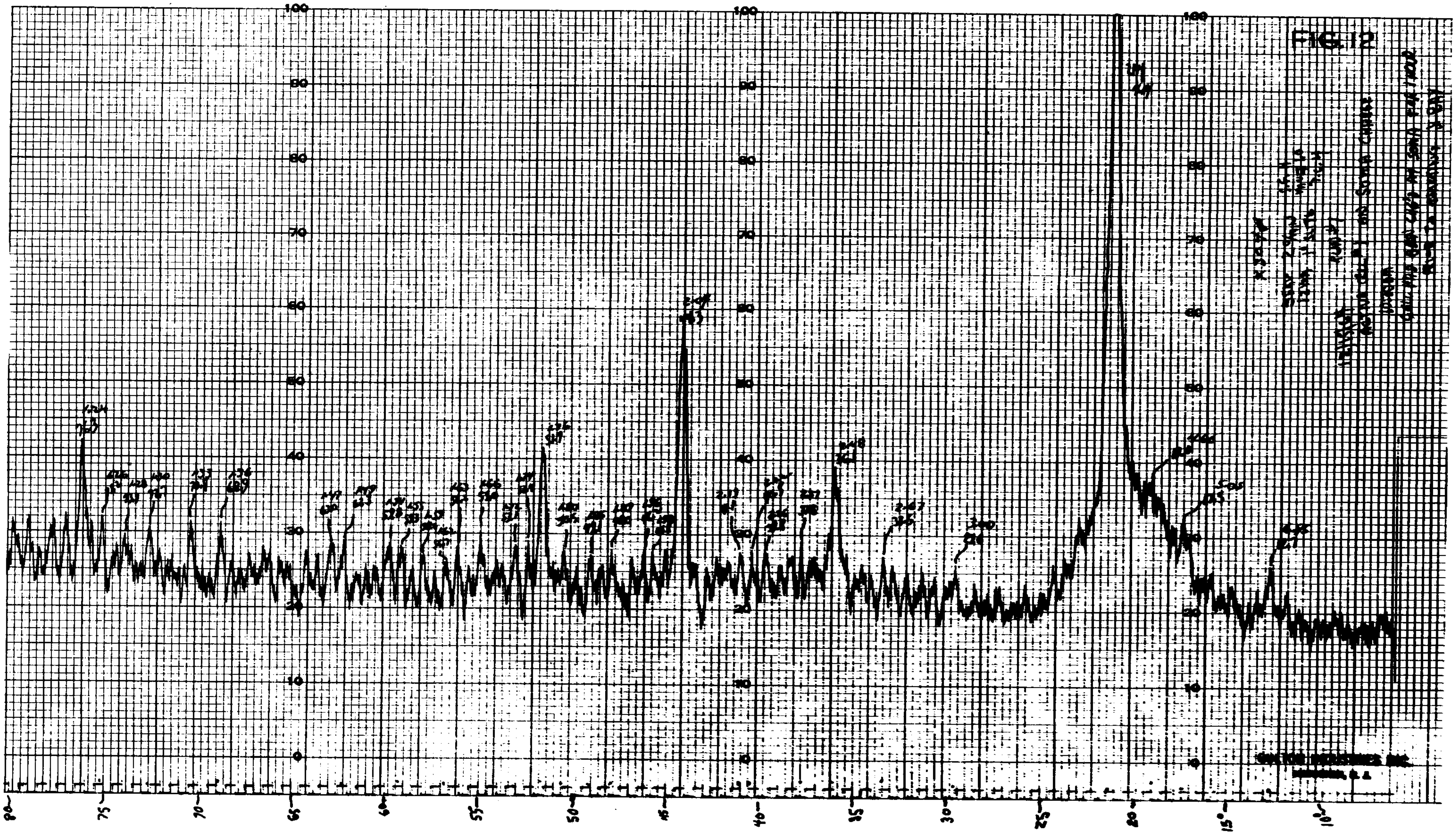


FIG. 12

FIG. 13

FIG. 14

FIG. 15

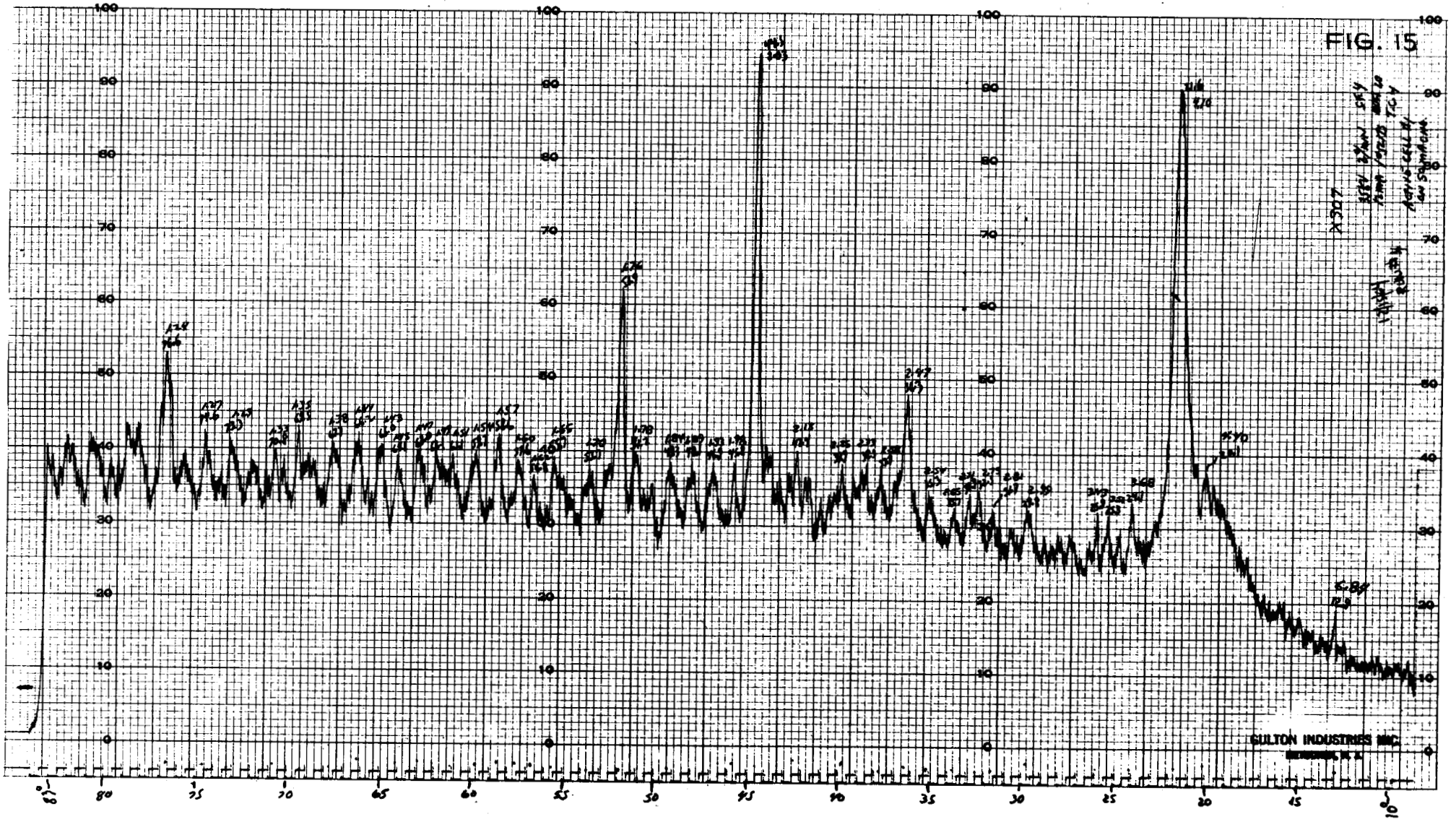


FIG. 16

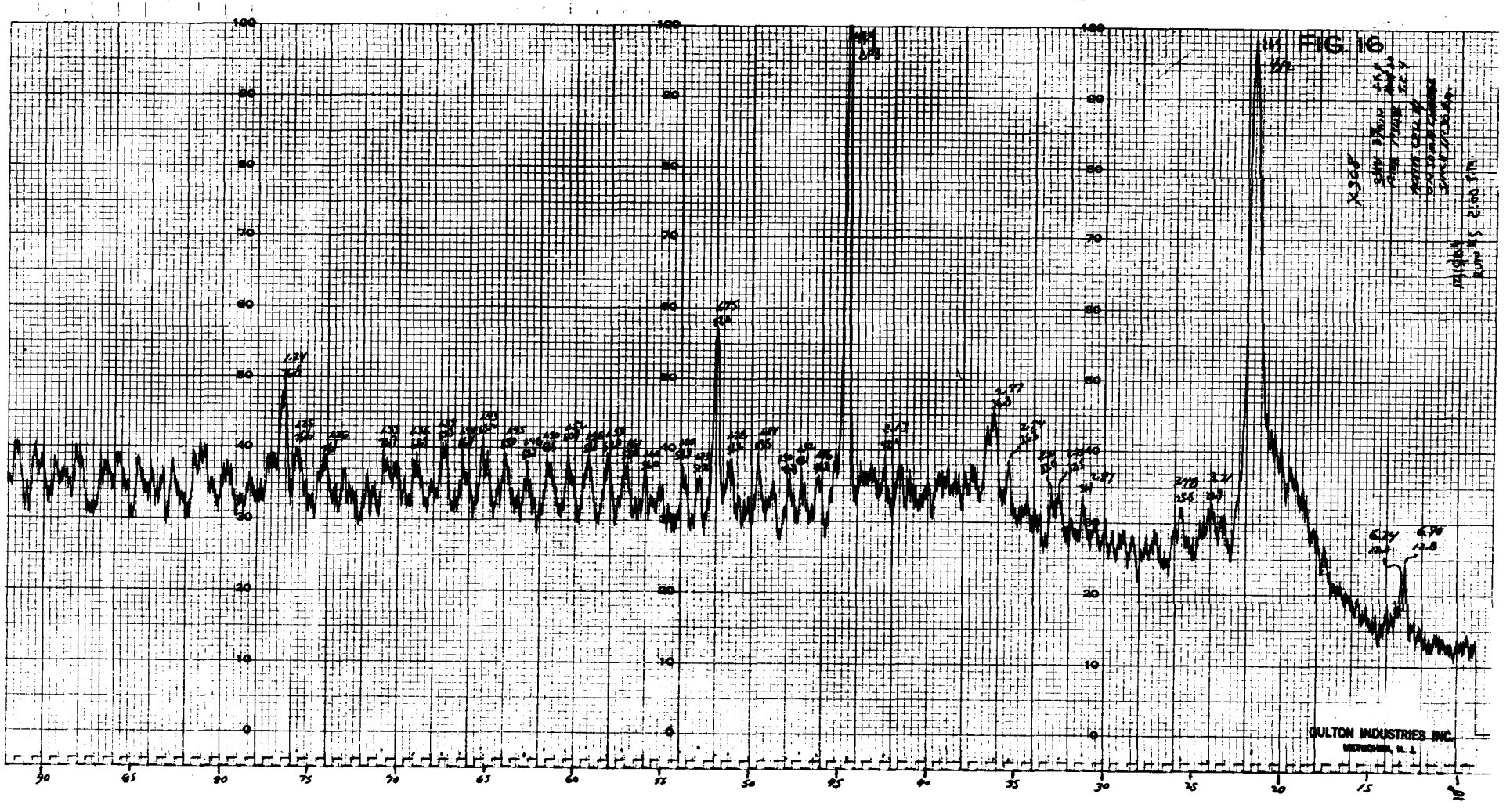


FIG. 17

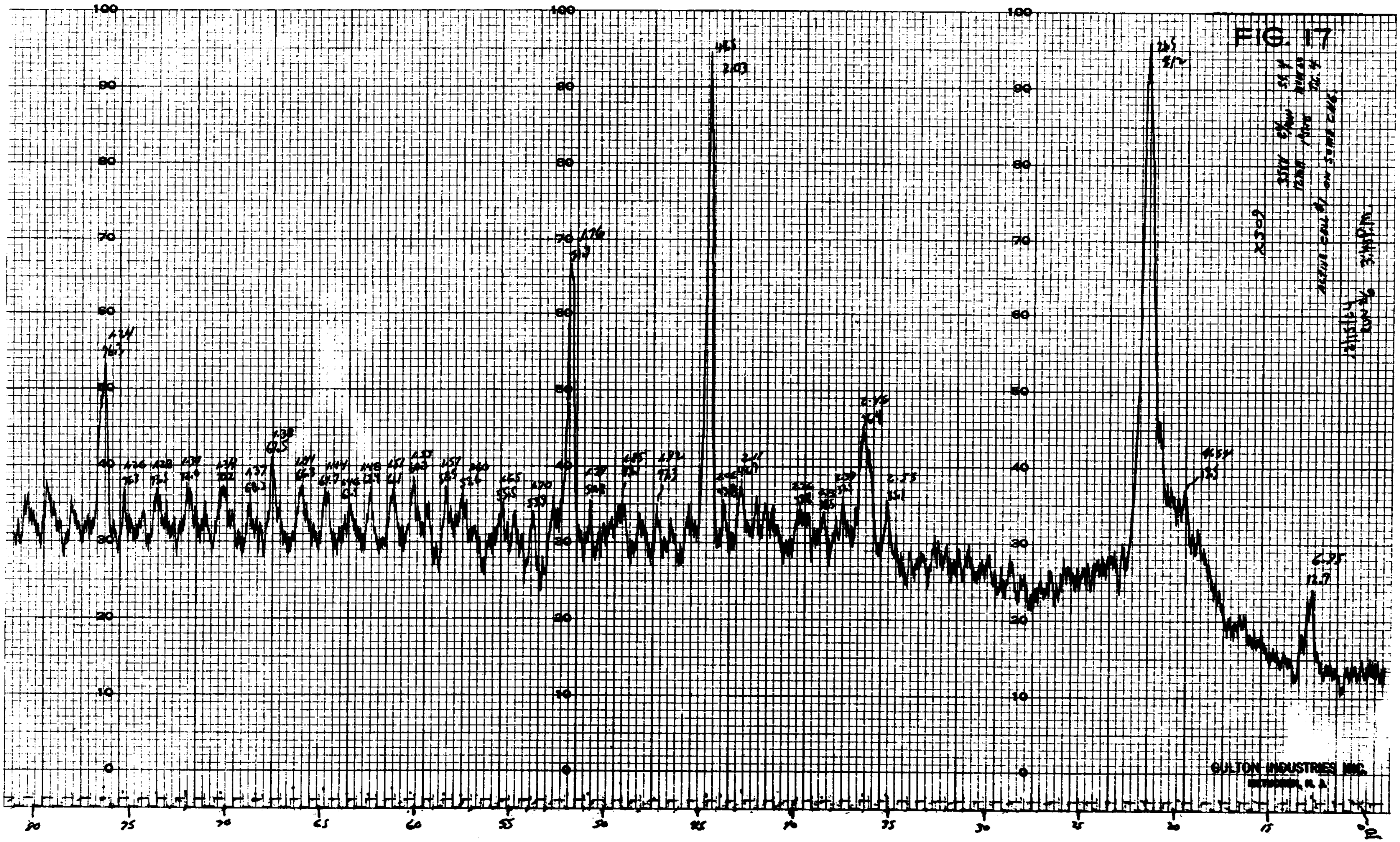


FIG. 18

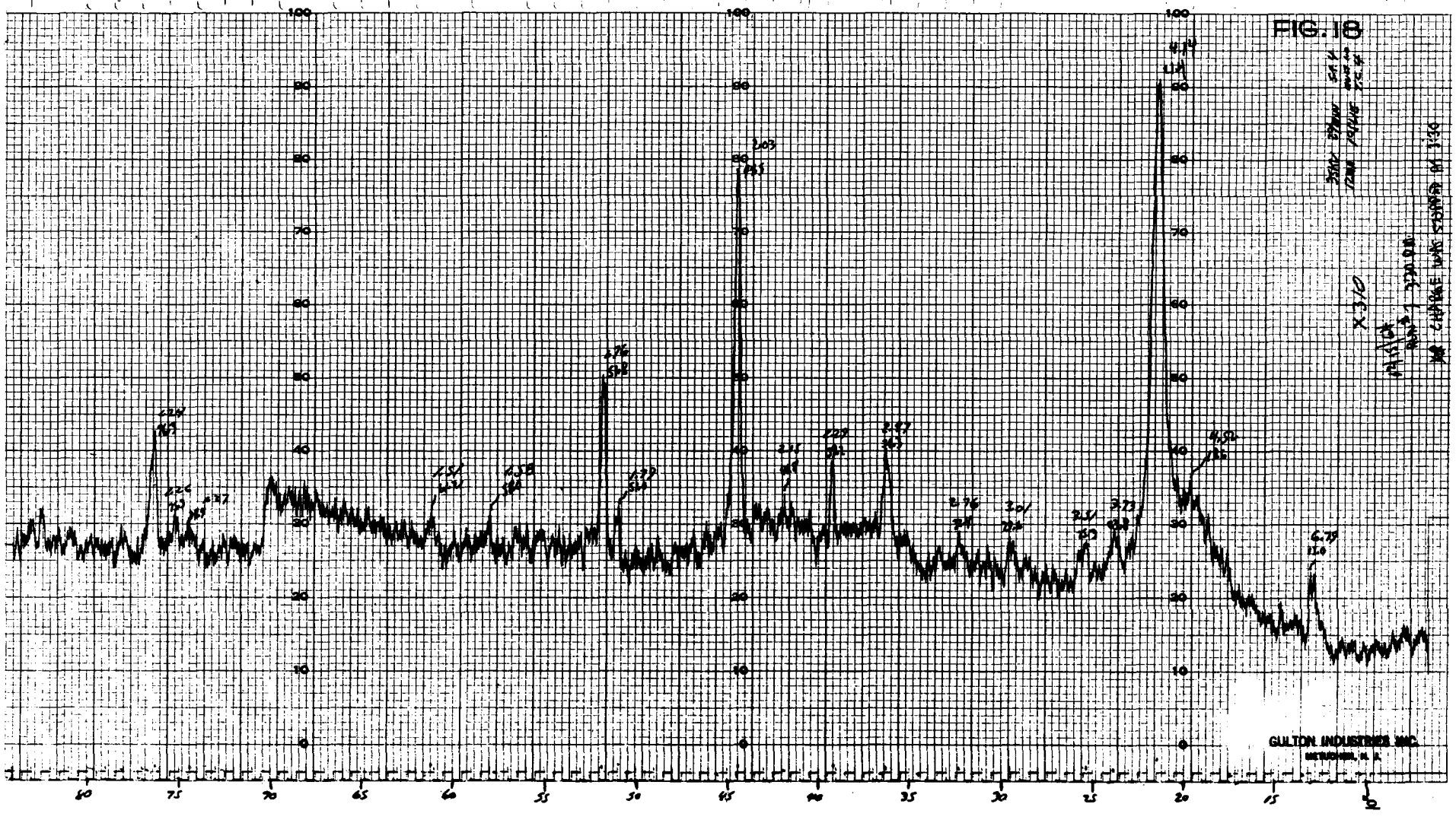


FIG. 19

FIG. 20

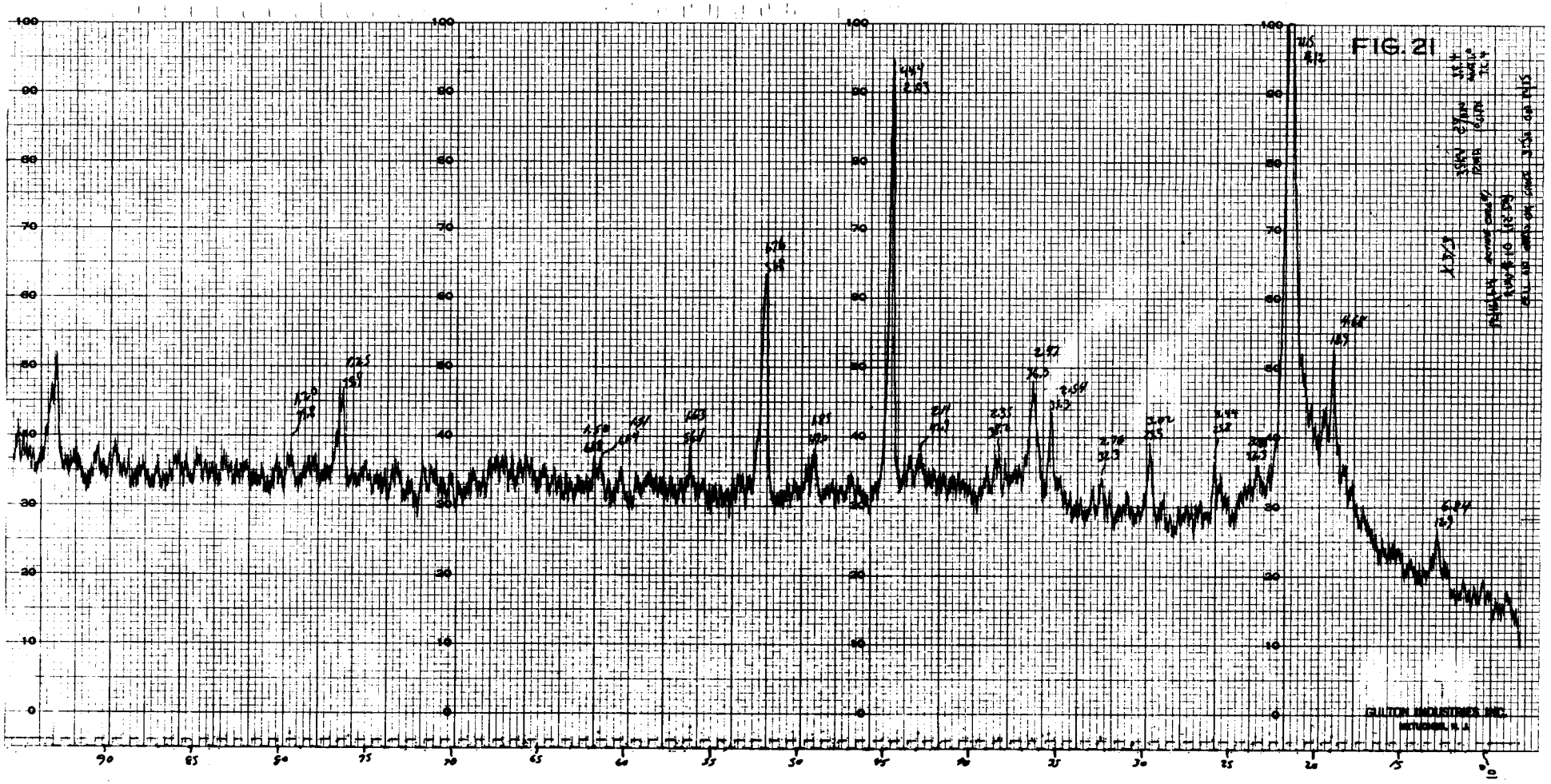


FIG. 21
GULTON INDUSTRIES, INC.

FIG. 22

FIG. 23

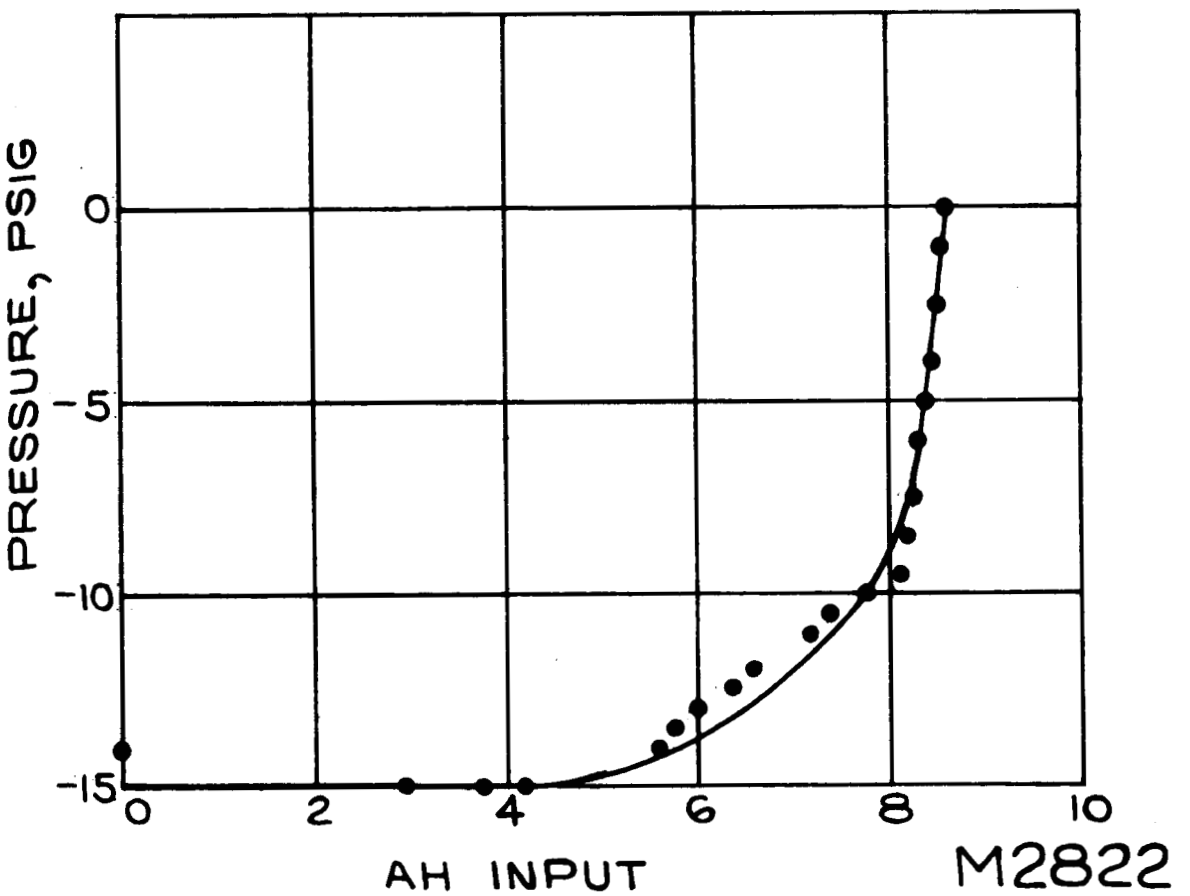


FIG. 24 FINAL SEALED-STARVED CHARGE

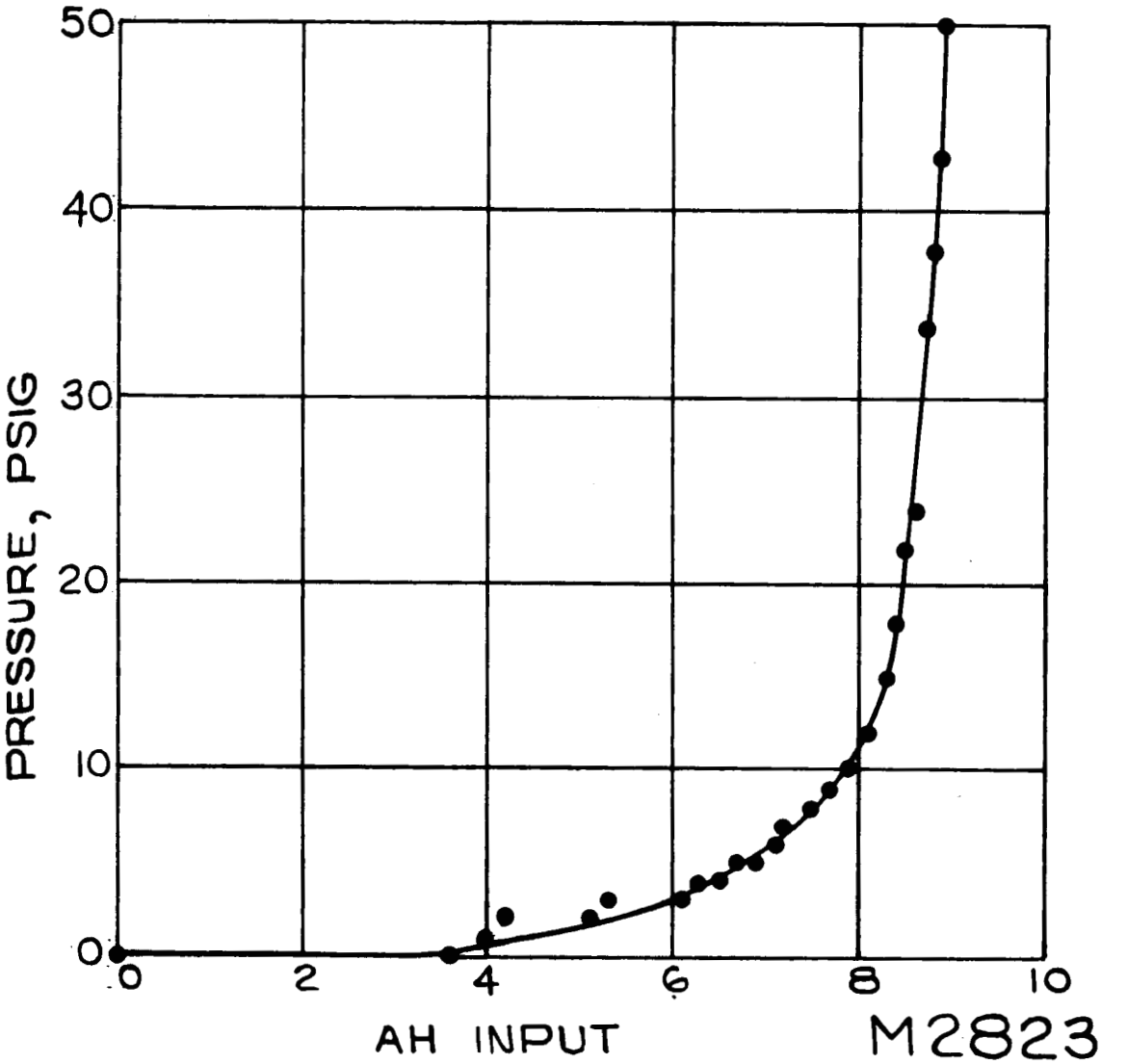


FIG. 25 FIRST FLOODED CHARGE

FIG 2
GULTON INDUSTRIES INC.