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# EFFECT OF CLEANING METHODS UPON ELECTROCHEMICAL BEHAVIOR OF HIGH-CARBON STEEL CATHODES IN A CHROMIC ACID ELECTROLYTE

BY

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# ABSTRACT

The quality of electrodeposited coatings is to a great extent determined by cleaning or surface-conditioning methods employed in preparing the basis metal for plating. Cleaning procedures are usually established empirically, by trial and error. Little or no experimental work was devoted to a quantitative study of measurable parameters that are influenced by the relative effectiveness of individual cleaning processes. The present experiment was designed in an attempt to establish a correlation between one such parameter (namely, the single-electrode potential of polarized cathode) and the quality of subsequent plating.

High-carbon steel cathodes, -a material that is often notoriously difficult to electroplate,- were used in the experiment. Each sample cathode was carried through one of 21 selected cleaning cycles, consisting of one or more individual steps. After cleaning, samples were placed into cell containing $CrO_3$ electrolyte and their potential was measured at various densities of polarizing current. When plotted in juxtaposition, all 21 Cathode Potential vs Current Density curves were found to posess a similarity of shape. Curves did, however, differ in their position with respect to the ordinate (used as the current density axis). The entire family of curves thus had the appearance of a band with a considerable and variable width in the direction of the abscissa. The possible significance of this horizontal shift was investigated by test-plating several samples. These samples were prepared by means of cleaning cycles that corresponded to most widely separated Potential-C.D. curves.

An examination of plated samples revealed that the quality of the deposit did, indeed, vary from sample to sample. The cleaning procedure corresponding to curve most distant from the ordinate produced sample surface that easily accepted the electrodeposit, while procedure corresponding to curve located near the ordinate produced a surface that was definitely more difficult to plate.

Thus, the experiment shows that 1) there is a one-to-one correlation between the cleaning method employed in preparing metal surface for plating and the quality of subsequent electrodeposit on this surface, 2) the single-electrode potential of the basis metal is a measurable parameter that can be used to evaluate the relative effectiveness of various metal cleaning methods, and 3) when plating chromium on polished hardened high-carbon steel, best results are obtained when basis metal is cleaned mechanically, by wet-scouring with a mildly abrasive slurry.

# FOREWORD

In the Fall of 1959, author was given a task of establishing a chromium plating procedure which was to be an important step in a salvaging operation involving some 15,000 critical sewing machine parts. The part in question was fabricated of AISI B-1113 Bessemer steel and was routinely case-hardened prior to plating; subsequent finishing operations consisted of grinding and polishing of exterior surfaces. Normally, plating of these parts causes no undue difficulty. However, 15,000-piece lot consisted mainly of parts which, due to variations in hardening operation, had an inferior case structure, characterized by unusually soft outer layer. To obtain a sound wear-resistant foundation for the chromium plate, this layer had to be removed by grinding.

The first few attempts to chromium plate parts from reworked lot produced an exceptionally high (up to 80%) reject percentage after plating. Plating defects comprised blistering, insufficient thickness, poor coverage, poor adhesion, dull deposit, etc. A closer inspection of rejected parts revealed that they could be divided into two groups, namely, 1) parts with a satisfactory plating on newly-ground surfaces and poor plating elsewhere, and 2) parts with poor plating on ground surfaces but with a good deposit on all other surfaces. Both these types of rejects, and parts with an entirely satisfactory plating as well, were found on any single plating rack, processed as a unit.

The ensuing investigation proceeded in the usual manner: bath composition and operating conditions were tested and necessary corrective changes made; current supply checked for an excessive A.C. ripple; plating racks stripped and cleaned to make a positive contact with parts and with the cathode bar; all anodes cleaned and re-positioned. Despite all these changes , however, the reject percentage remained at a high level.

The fact that changes made at the plating tank failed to produce an improvement indicated that a non-uniform surface condition was created on parts in the course of pre-plating treatment and that an investigation of the cleaning schedule was in order.

A few exploratory changes were made in the cleaning cycle but this produced no noticeable lowering of the percentage of rejects. A literature search and inquiries amongst platers revealed similar experiences but no agreement as to the cause or cure of this problem, indicating that only a lengthy study could possibly provide a solution.Tight production schedule, however, did not allow enough time for a basic investigation. The problem of plating the difficult 15,000-piece lot of parts was therefore solved by a "shotgun" approach.A cleaning cycle was arbitrarily selected and parts were processed accordingly. Poorly plated parts were separated by a 100% inspection, stripped of chromium, cleaned mechanically by vapor-blasting, and then processed again. This treatment reduced the overall reject percentage to approximately 10%. Parts that failed to take a satisfactory plate during the second run were scrapped.

While the above solution appeared satisfactory from production viewpoint, author decided to continue the study of the cleaning cycle as a matter of academic interest. The method and the results of this investigation are discussed in subsequent sections of the present paper.

# INTRODUCTION

The quality of electrodeposited metallic coatings and particularly their adhesion to the basis metal is greatly affected by the surface condition of the latter (11, 18, 20, 42, 59, 63, 68) *). The surface condition, in turn, is influenced by numerous factors, such as the nature of the basis metal (5, 11, 45), the type of heat treatment received by the part (5, 40), the mechanical methods and techniques used in manufacturing the part (32, 34, 35, 36), as well as the mechanical, chemical, or electrochemical pre-plating treatment (5, 34, 39, 48, 59, 63).

The first three factors are usually selected on the basis of functional considerations and can only rarely be changed to suit a plater's purpose. The last factor, however, - commonly referred to as "cleaning",- is entirely within the plater's domain and can be varied to suit specific plating conditions.

The importance of cleaning was known years ago and cleaning methods have been the subject of attention of numerous investigators.(5, 24, 29, 39, 42, 46, 48, 59, 63, 68, 71, 72, 76). Many have stressed the fact that cleaning requirements are most exacting when a metal is prepared for chromium plating (5, 17). Cleaning becomes even more important when hardened high-carbon or carburized steel is to be chromium plated (35, 40, 48). In the latter case, however, research was concentrated on the problem of finding a suitable pre-plating treatment for "tool chromium" rather than for "ornamental chromium" plating.

While studying various metal-cleaning techniques, most in-

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*) See bibliography.

vestigators employed somewhat subjective methods of evaluation of surface cleanliness, such as the atomizer test or the water-break test. In some cases, cleaning cycles were rated on the basis of a performance test, i.e., cleaned parts were plated under controlled conditions and the quality of resultant plate was the basis for evaluation of the effectiveness of the pre-plating cleaning cycle (38, 39, 50, 69). Still, more often than not, the quality of the plate was judged on a subjective basis of gloss, appearance, etc.

Several investigators of cleaning methods expressed an opinion that in the case of plating chromium over steel many seemingly unexplainable difficulties are, perhaps, due to the fact that some cleaning methods tend to lower the overvoltage of hydrogen (16, 35, 45, 57, 65), thus reducing cathode efficiency in an unpredictable manner. It appears that in localized areas of the cathode the efficiency is sometimes reduced almost to zero.

The overvoltage lowering theory, however, is largely presented as a speculation and is seldom supported by experimental numerical data. This statement should not be read to mean that no research was devoted to hydrogen overvoltage. On the contrary, numerous papers dealing with this subject have been published (26, 28, 30, 32, 33, 35, 41, 43, 49, 55, 57, 60, 61, 71).

In most cases, however, authors of overvoltage papers are interested mainly in the theoretical aspect of the subject. Yet, they established that hydrogen overvoltage is affected by various factors in the following manner. The overvoltage increases with the increasing density of polarizing current and, when the current density becomes sufficiently high, it tends to approach a limiting value of about 1.32 volts (10).At this point the magnitude of overvoltage is not dependent on the cathode material. Overvoltage varies inversely with the electrolyte temperature and decreases slightly when pH of the solution increases;its magnitude is affected even by traces of catalytic poisons and by the rate of $H_2$ diffusion through the cathode (21).Overvoltage of hydrogen is higher when certain plating bath additives are present or when the metal ion content of the bath is lowered. When superimposed A.C. or chemical depolarizers are present, a lower hydrogen overvoltage is observed(8, 11).

Overvoltage experiments described in the majority of papers were usually performed under idealized conditions. Electrodes were often made of platinum, electrolytes were saturated usually with inert gases and their concentration kept well below that commonly encountered in plating solutions. Only rarely does a researcher use an actual plating solution for an overvoltage or a polarization study (19, 78, 79).

It is difficult, if not impossible, to extrapolate data collected under idealized conditions to those encountered within a plating tank,especially a plating tank containing chromium bath. Rather than make an attempt to correlate data from various overvoltage studies with plating room difficulties, author decided to conduct an experiment which would show, in a measurable way, how does the pre-plating treatment affect the electrodeposition of chromium on high-carbon steel. The author also expected to find, as a by-product of the experiment, a simple objective method that could be used for evaluation of the relative effectiveness of metal-cleaning techniques.

A detailed description of the experimental set-up and procedure will be given elsewhere in this paper. At this point it should suffice to state that the experiment consisted of:

1. Selecting several cleaning schedules, preferably resembling those used in plating industry.
2. Processing multiplicate samples in accordance with these schedules.
3. Measuring cathode potential against a reference electrode at various densities of the polarizing current.
4. Constructing a "Polarized Current vs Cathode Potential" curve for each group of samples pre-treated in accordance with one specific cleaning schedule.
5. Concluding, on the basis of shapes or relative location of current-potential curves, which cleaning schedule will most likely result in a best or in a poorest plating quality.
6. Confirming or disproving this conclusion by test comprising an actual plating of several samples treated in accordance with cleaning schedules selected in Step 5.

It should be noted here in reference to Step 1, that no acid "dips" of short duration were employed. While such dips are widely used by platers as final or activating step of a cleaning schedule, the author felt that they will be difficult to duplicate and thus would only contribute to ambiguity. Similarly, no wetting agent was used in water rinses. There are indications that the presence of certain organic substances in an electrolyte has a tendency to cause variations in hydrogen overvoltage (41); author feared that wetting agents, when dragged into the electrolysis cell, might produce a similar effect.

Methods used for measuring the hydrogen overvoltage or potential of an electrode are quite widely discussed in literature (2, 4, 8, 9, 10, 11, 13, 18, 21, 26, 28, 31, 43, 45, 49, 57, 60, 61, 67) and range from the very simple, such as proposed by Haring (20, 43), to the very elaborate, designed with the purpose of reducing the number of experimental variables to an absolute minimum (61, 67).

Essentially, all overvoltage studies employ modifications of but two basic methods, namely, the direct method and the commutator method of measurement. When the former is used, both the reference electrode and the polarizing current source are tied permanently to the electrode being investigated; the potential of this electrode is measured while the current is on. In the latter method, a provision is made to connect reference electrode to the electrode being investigated only when the polarizing current is switched off. The relative merits of both methods are discussed at length in literature (4, 9, 10, 11, 12, 21, 60),but it seems that in the hands of skilled investigator either method is capable of producing satisfactory results, - at least, when used in conjunction with a low-concentration electrolyte.

For the present work, the direct method of measurement was selected as a matter of convenience. A trial run of a mechanical commutator revealed that meter reading is greatly influenced by the conditions of the commutator surfaces, as well as by the fluctuations in the switching rate. The use of an electronic switching device would, of course, eliminate both factors, but would also complicate the instrumentation unnecessarily.

The simple Haring's method (43), based on the use of a reference electrode made of the same material as the electrode being investigated, was very attractive. However, only a mild attempt was made to utilize this approach. Author feared that, although the overvoltage calculated for various samples would always be the difference of potential between the polarized and the non-polarized metal, the comparison of overvoltages of samples cleaned by different methods would not be valid. Later in the experiment, this assumption was confirmed.The uniformity of the sample surface conditions varied within every group of multiplicate samples, the magnitude of these variations being dependent upon the cleaning schedule employed.

Haring's method was abandoned in favor of a direct method designed around a saturated calomel reference electrode and a vacuum-tube voltmeter. The latter instrument, rather than the more conventional potentiometer, was selected for the following reasons. It is commonly assumed that a potentiometer or a bridge circuit draws no current from the circuit under measurement. This is, however, true only after the circuit is brought to balance. The necessary "tapping" during the balancing operation causes a small and indeterminable current draw which might or might not have an effect upon the results of the measurement. A sensitive potentiometer would also indicate potential changes as small as one millivolt and thus its readings might reflect even the random fluctuations of the potential. On the other hand, a vacuum-tube voltmeter with its inherently high input resistance (upward from 10 - 20 megohms) draws a constant but small current, and would affect all measurements to same degree. A vacuum-tube voltmeter of the type used in this experiment cannot be read closer than 10-20mV and thus, being a relatively "blunt" instrument, reacts only to permanent changes in the electrode potential.

The last item to be considered in setting up the experiment was the electrolyte. As previously explained, the experiment was to be conducted under conditions approximating those found in a plating tank. Therefore, a commercial chromium plating formulation was selected. Since, however, a full-strength plating bath contains at least 250 g/l of $CrO_3$ and is, therefore, rather opaque, experimental electrolyte was diluted until it attained a degree of transparency which permitted a continuous visual observation of the sample surface.

# EXPERIMENTAL PROCEDURE

1. Place the reference electrode assembly (Fig. 5) into its clamp on the electrode carrier (Fig.3); tighten the clamp.
2. Secure the electrode carrier in a topmost position at the support rod. Attach flexible leads of the carrier to appropriate terminals of the polarizing and measuring circuit .
3. Assemble the electrocleaning setup.
4. Fill the electrolysis cell with electrolyte and lower the stirrer into solution. Clamp flexible shaft of the stirrer to the support rod.
5. Insert an anode plate into its holder. Wrap stop-off tape around the plate so that its lower edge is 13 mm above the bottom end of the anode(Fig.4). Insert terminal pin of the anode holder into the appropriate clamp of the electrode carrier, making sure that the locating pin of the anode holder enters the locating slot in the carrier arm (Fig.3).
6. Polish the surface of sample with sandpaper, first lengthwise, then by rotating the sample between two pieces of sandpaper.
7. Insert polished sample into the cathode holder. Slide a PVC masking-off sleeve onto the sample so that its lower edge is 13 mm above the bottom end of the sample.
8. Process the sample in accordance with one of cleaning schedules (see list). After the last rinse, shake off excess

water and quickly insert the terminal pin of the cathode holder into the appropriate clamp of the electrode carrier, making sure that the locating pin of the holder enters the locating slot of the carrier arm (Fig.3) and that the sample does not come in contact with the tip of the reference electrode assembly.

9. Place electrodes into the electrolysis cell by moving the electrode carrier to its lowest position on the support rod. Start the stirrer.

10. Read cathode potential on the scale of vacuum-tube voltmeter ($M_2$ in Fig.1) and record this reading.

11. Switch on the polarizing current and adjust it to 0.5mA by means of the potentiometer and milliammeter (respectively, $R_1$ and $M_1$ in Fig.1 ). Read and record the cathode potential.

12. Repeat Step 11, adjusting polarizing current to 1, 2, 3, 4, 5, 6, 8, 10, 15, 20, 25, 30, 35, 40, 45, and 50 mA.

13. Switch off the polarizing current and take the electrode out of the electrolysis cell by shifting the electrode carrier to its topmost position on the support rod. Stop the stirrer.

14. Remove the cathode holder from its clamp. Rinse sample clean, remove from sample holder, and label it with the appropriate cleaning schedule number.

15. Repeat Steps 6 through 14, until a triplicate run is made for every cleaning schedule listed.

16. Plot cathode potential versus polarizing current, using abscisa axis for the former.

17. Visually, compare shape and relative positions of all plotted curves. Select two curves that are least similar and one that is approximately half-way between these two.

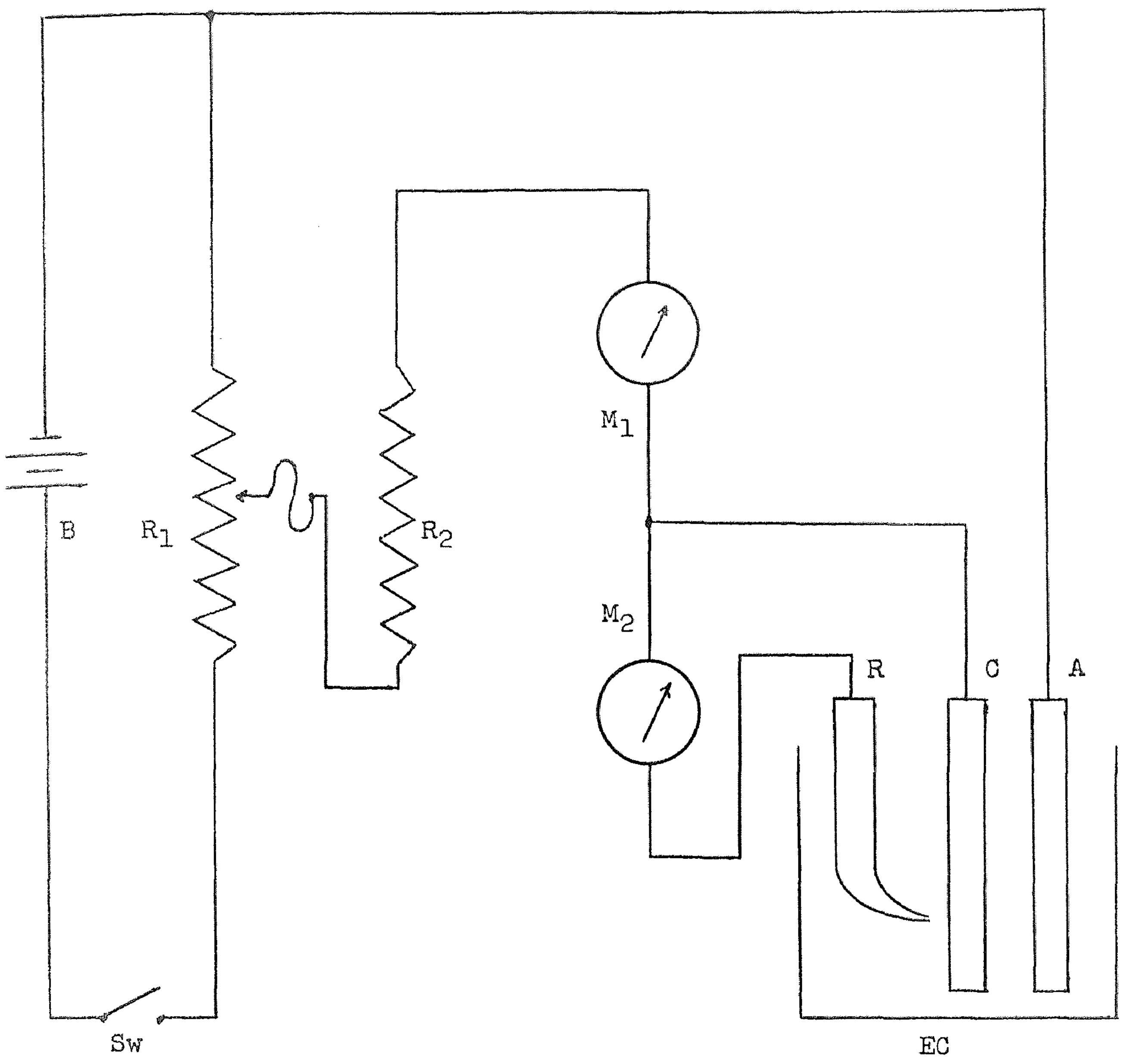
18. Process triplicate samples in accordance with cleaning schedules corresponding to the three curves selected in step 17 and electroplate these in a bath containing 250 g/l of CR-110. Plate each sample for 5.0 minutes at 45°C and 2.0 amp/sq.in.

19. Weigh plated samples to the nearest 0.1 mg and record the weight. Strip chromium, by making samples anodic in the electrocleaning bath, for 3.0 minutes at 100asf.

20. Rinse samples, dry, and re-weigh. Calculate weight of the chromium deposit.

21. From the weight of chromium and the apparent area of the sample, calculate the thickness of the deposit.

Fig. 1 POLARIZING CIRCUIT



B - battery, 12 v

$R_1$- potentiometer, 1,000Ω , 2 w

$R_2$- resistor, 150Ω , 2 w

Sw- on-off switch, S.P.S.T.

$M_1$- milliammeter

$M_2$- vacuum tube voltmeter

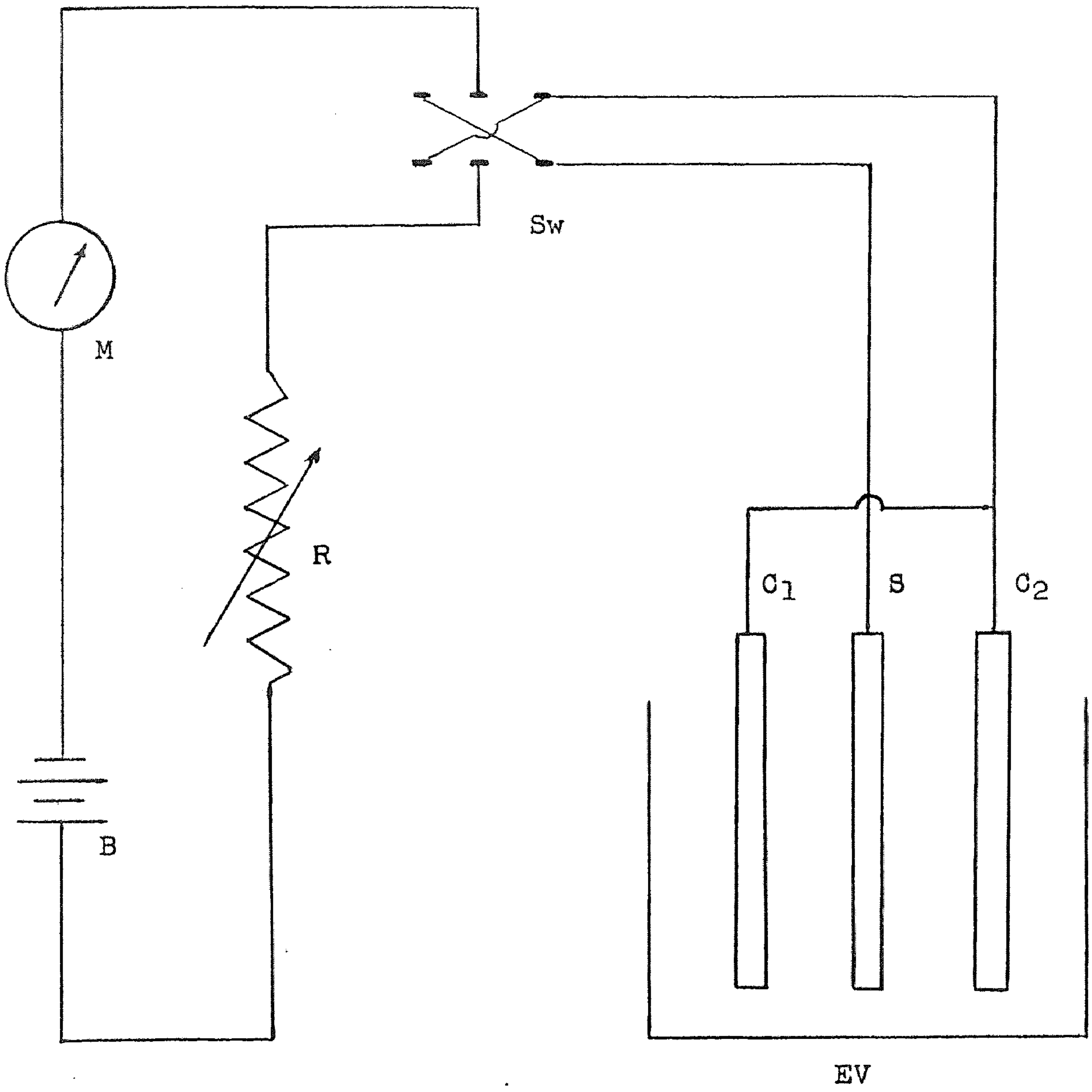
EC- electrolysis cell

R - reference electrode

C - cathode

A - anode

Fig. 2 ELECTROLYTIC CLEANING CIRCUIT



B - battery, 6 v

R - rheostat, 600Ω , 2 w

M - milliammeter

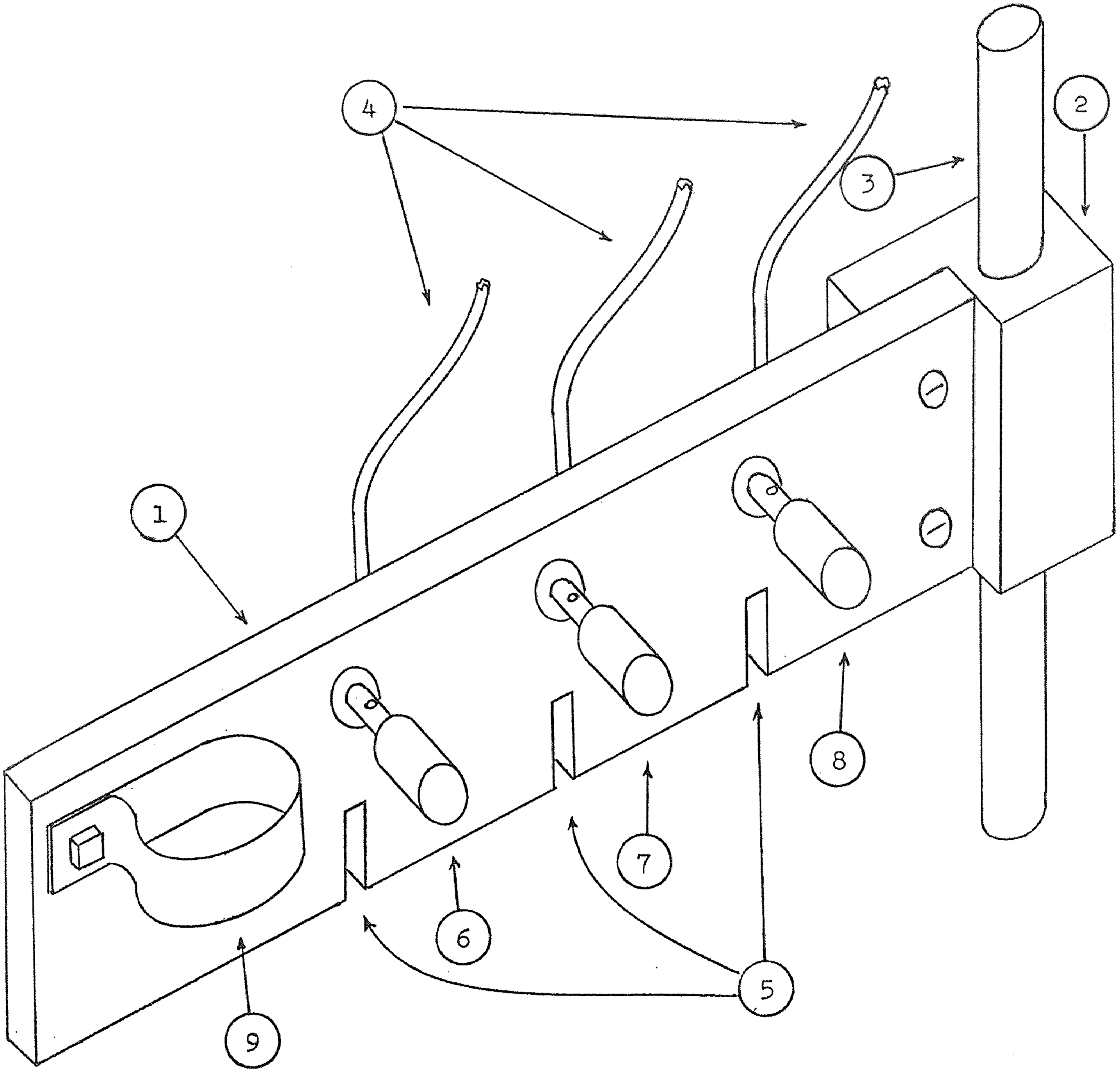
Sw - on-off and polarity reversing switch, D.P.D.T.

EV - electrolyte vessel

S - sample to be cleaned

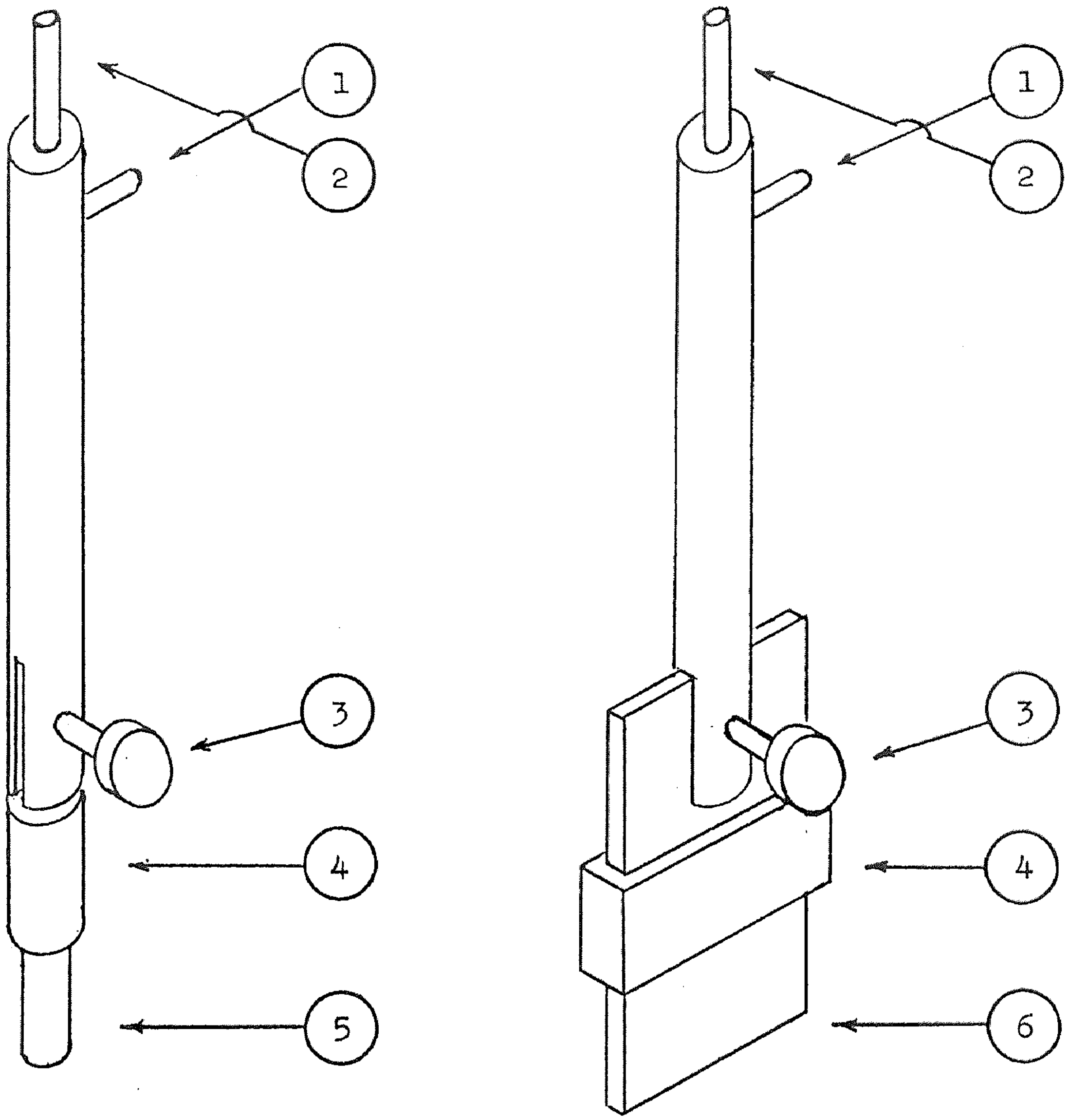
$C_1$, $C_2$ - counter-electrodes

Fig. 3 ELECTRODE CARRIER



1 - carrier arm, plexiglass
2 - support clamp, aluminum
3 - support rod, aluminum
4 - flexible leads
5 - locating slots
6 - cathode terminal and clamp
7 - anode terminal and clamp
8 - reference electrode terminal
9 - reference electrode clamp

Fig. 4 <u>ELECTRODE HOLDERS</u>



Cathode

Anode

1 - locating pin

2 - terminal pin

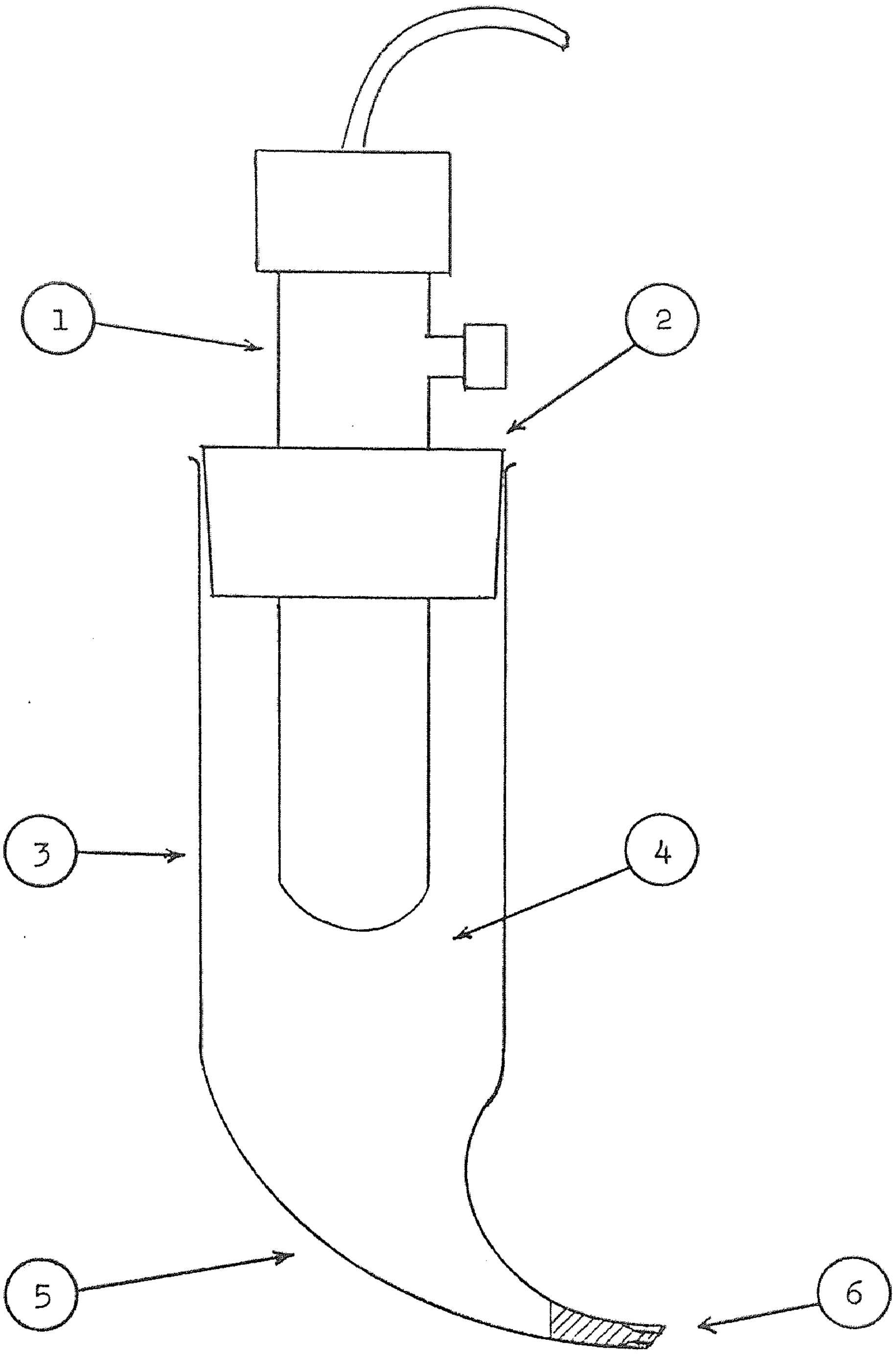
3 - set screw

4 - stop-off sleeve, PVC

5 - sample

6 - anode plate

Fig. 5 REFERENCE ELECTRODE ASSEMBLY



1 - sat. calomel electrode

2 - rubber stopper

3 - glass jacket

4 - sat. KCl solution

5 - bridge arm, plugged

6 - capillary tip

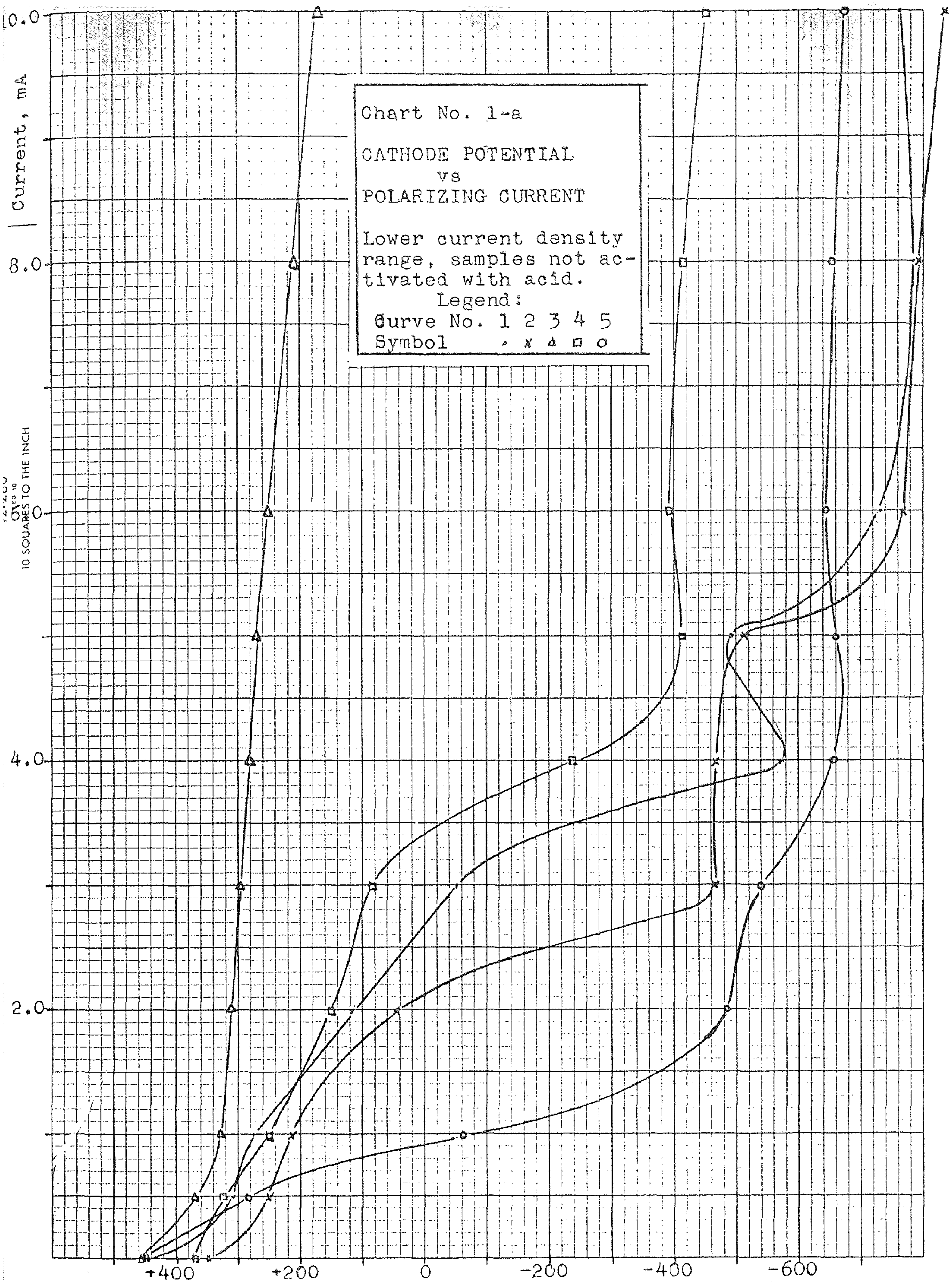


Chart No. 1-a
CATHODE POTENTIAL
vs
POLARIZING CURRENT
Lower current density range, samples not activated with acid.
Legend:
Curve No. 1 2 3 4 5
Symbol · x △ □ o
Current, mA
10.0
8.0
6.0
4.0
2.0
+400
+200
0
-200
-400
-600
Cathode potential, mV
10 SQUARES TO THE INCH

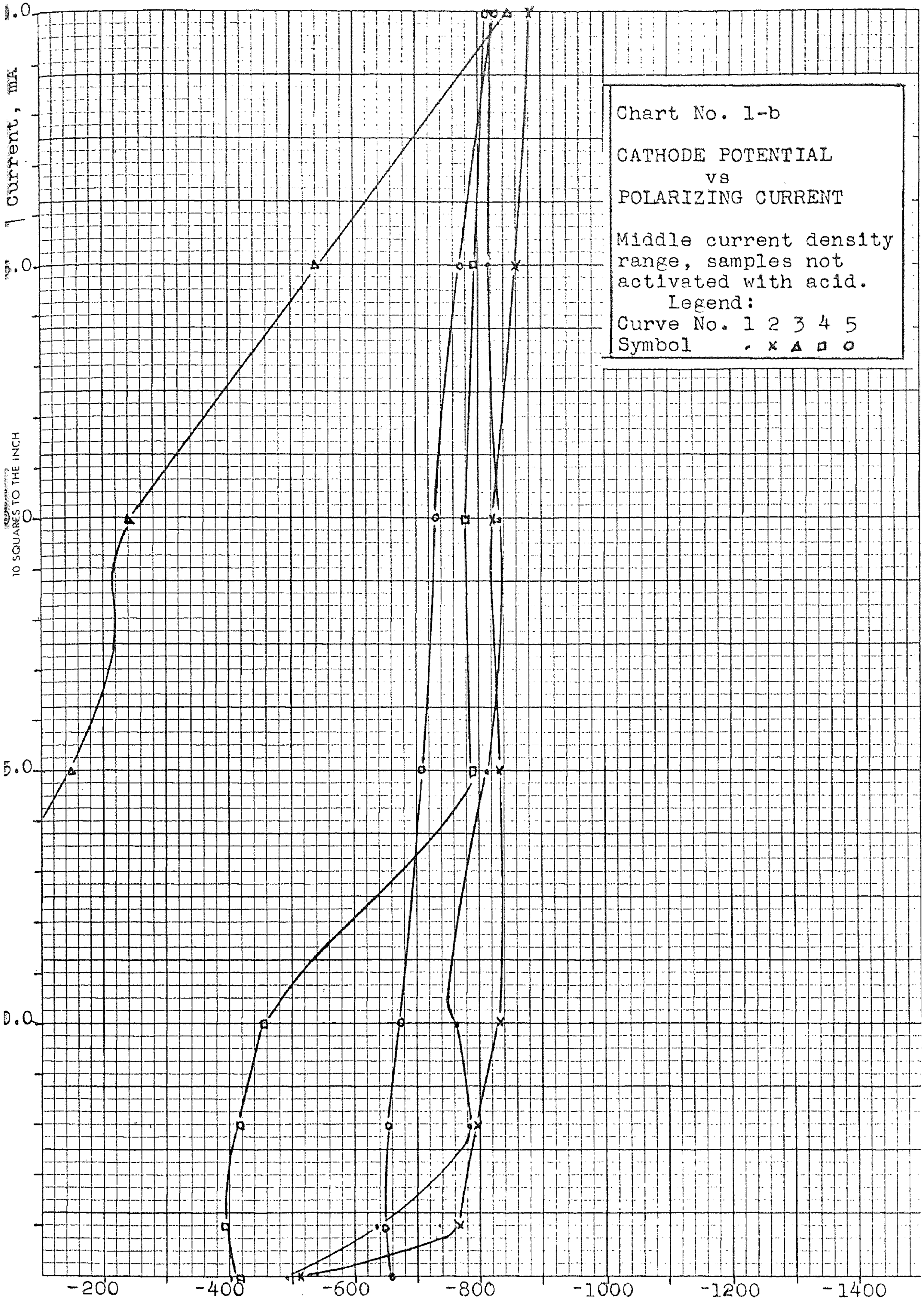


Chart No. 1-b

CATHODE POTENTIAL
vs
POLARIZING CURRENT

Middle current density range, samples not activated with acid.

Legend:

| Curve No. | 1 | 2 | 3 | 4 | 5 |
|---|---|---|---|---|---|
| Symbol | • | × | Δ | □ | ○ |

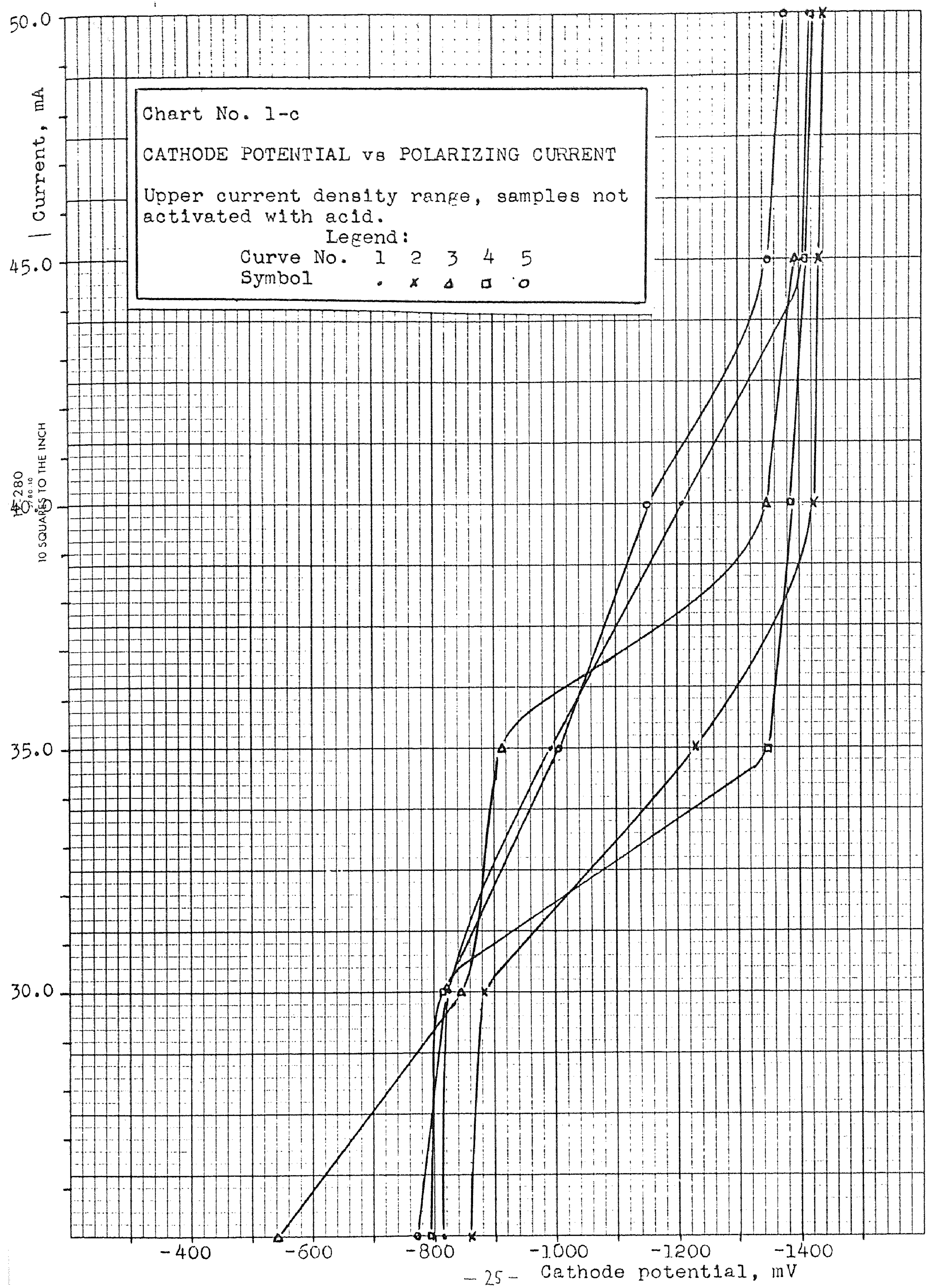


Chart No. 1-c

CATHODE POTENTIAL vs POLARIZING CURRENT

Upper current density range, samples not activated with acid.

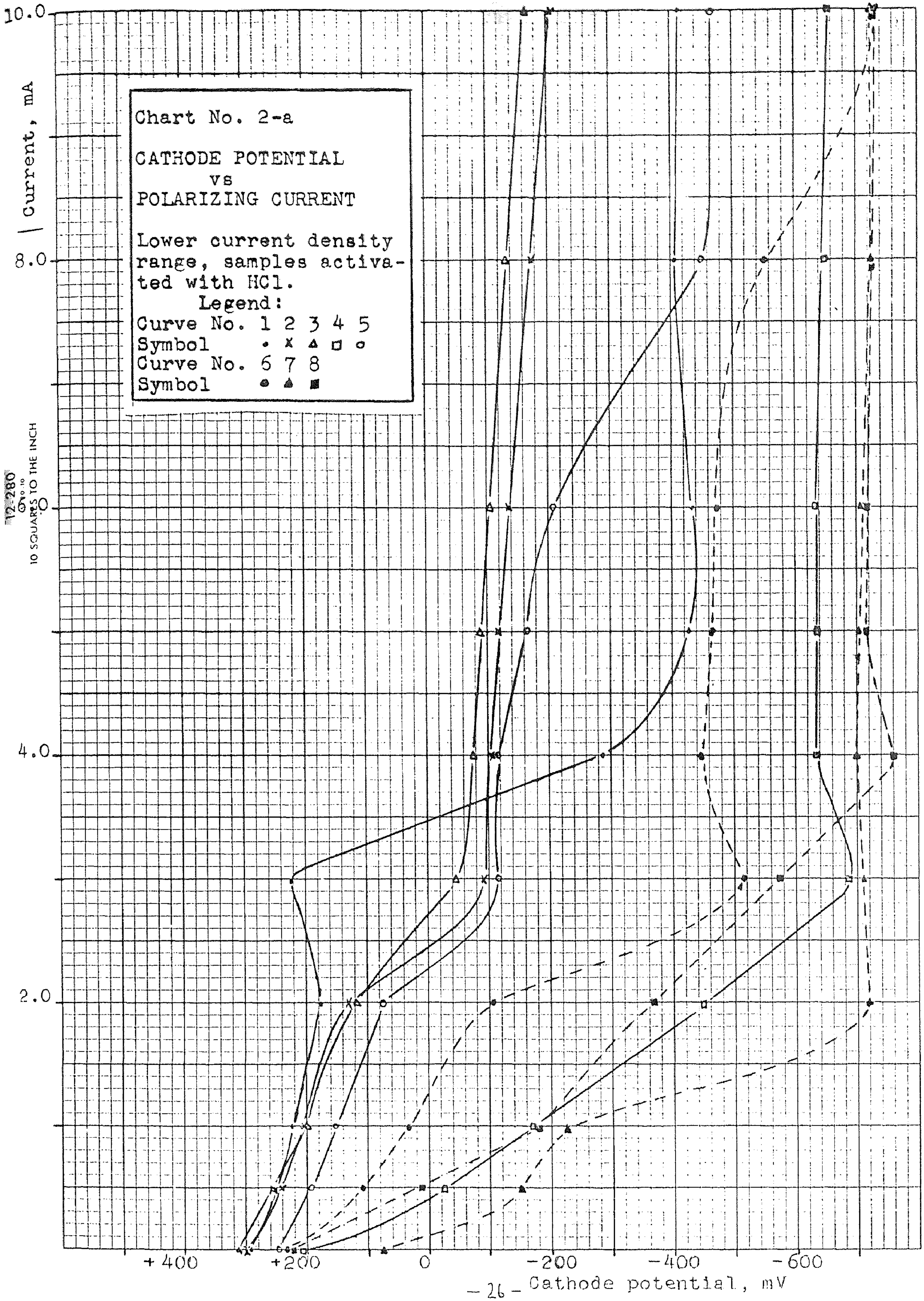


Chart No. 2-a

CATHODE POTENTIAL
vs
POLARIZING CURRENT

Lower current density range, samples activated with HCl.

Legend:

| Curve No. | 1 | 2 | 3 | 4 | 5 |
|---|---|---|---|---|---|
| Symbol | • | × | △ | □ | ○ |

| Curve No. | 6 | 7 | 8 |
|---|---|---|---|
| Symbol | ● | ▲ | ■ |

12 280
10 SQUARES TO THE INCH

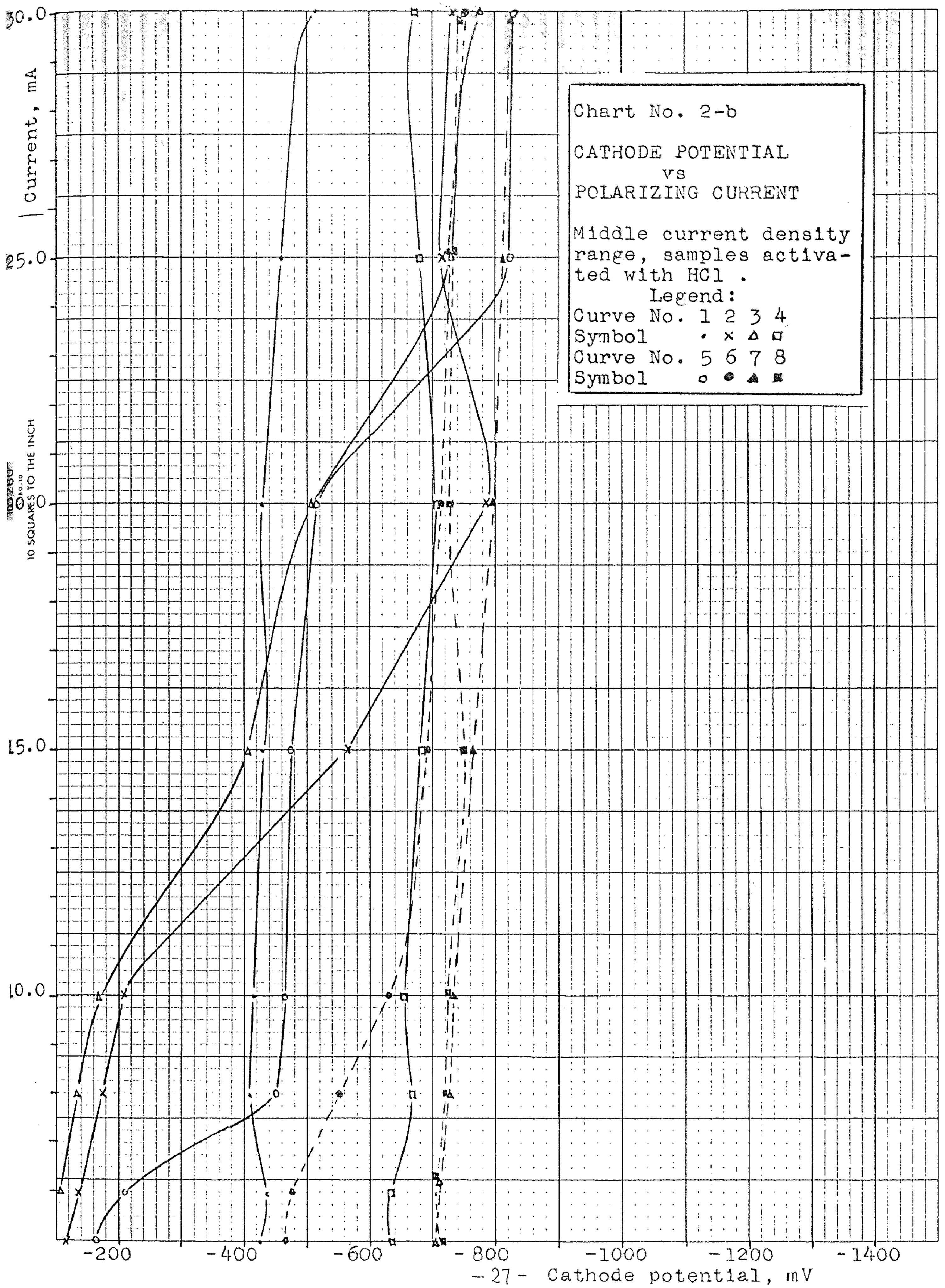


Chart No. 2-b

CATHODE POTENTIAL
vs
POLARIZING CURRENT

Middle current density range, samples activated with HCl .

Legend:

| Curve No. | 1 | 2 | 3 | 4 |
|---|---|---|---|---|
| Symbol | · | × | △ | □ |
| Curve No. | 5 | 6 | 7 | 8 |
| Symbol | ○ | ● | ▲ | ■ |

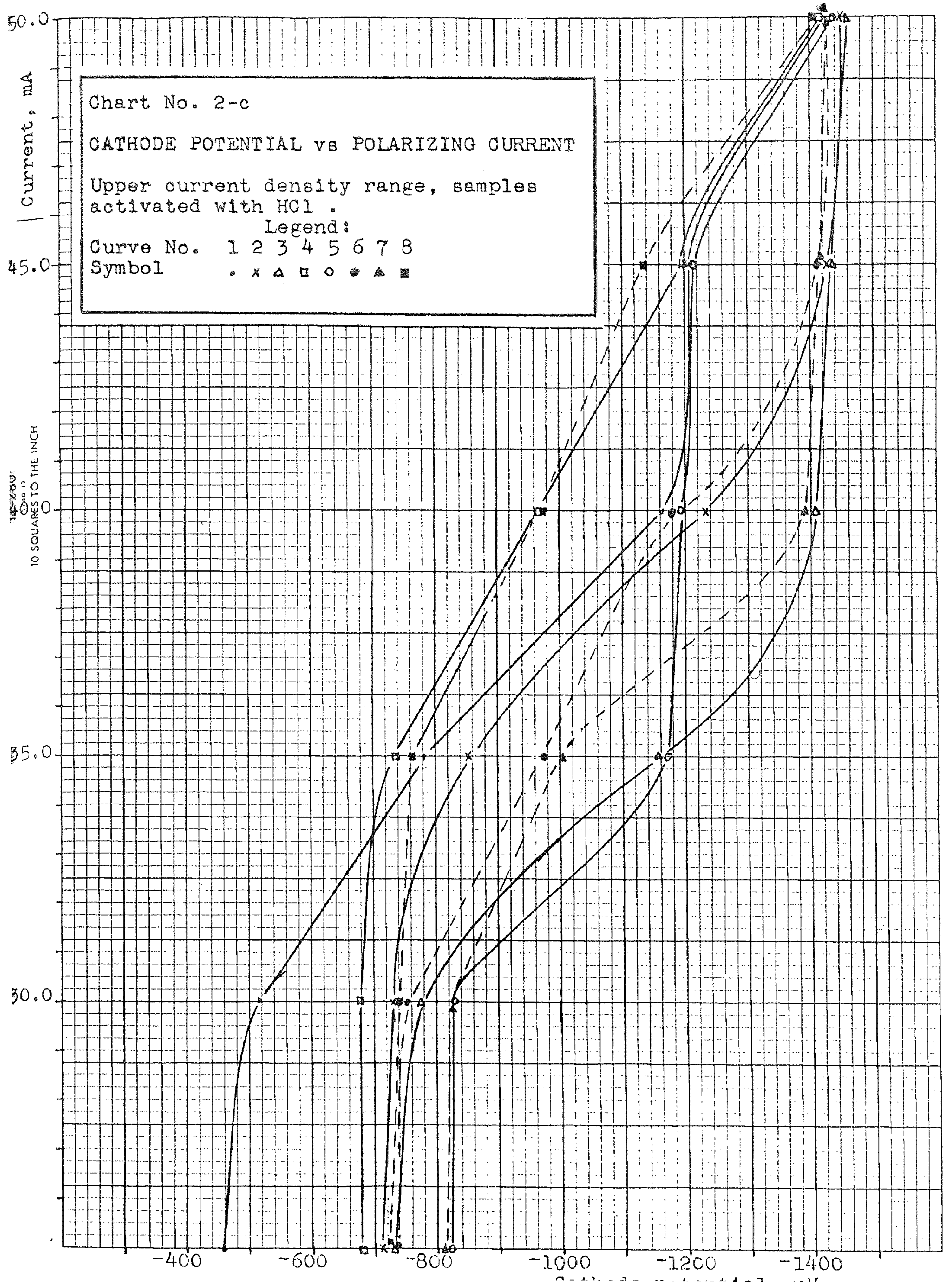


Chart No. 2-c

CATHODE POTENTIAL vs POLARIZING CURRENT

Upper current density range, samples activated with HCl .

Legend:

| Curve No. | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 |
|---|---|---|---|---|---|---|---|---|
| Symbol | · | × | △ | □ | ○ | ● | ▲ | ■ |

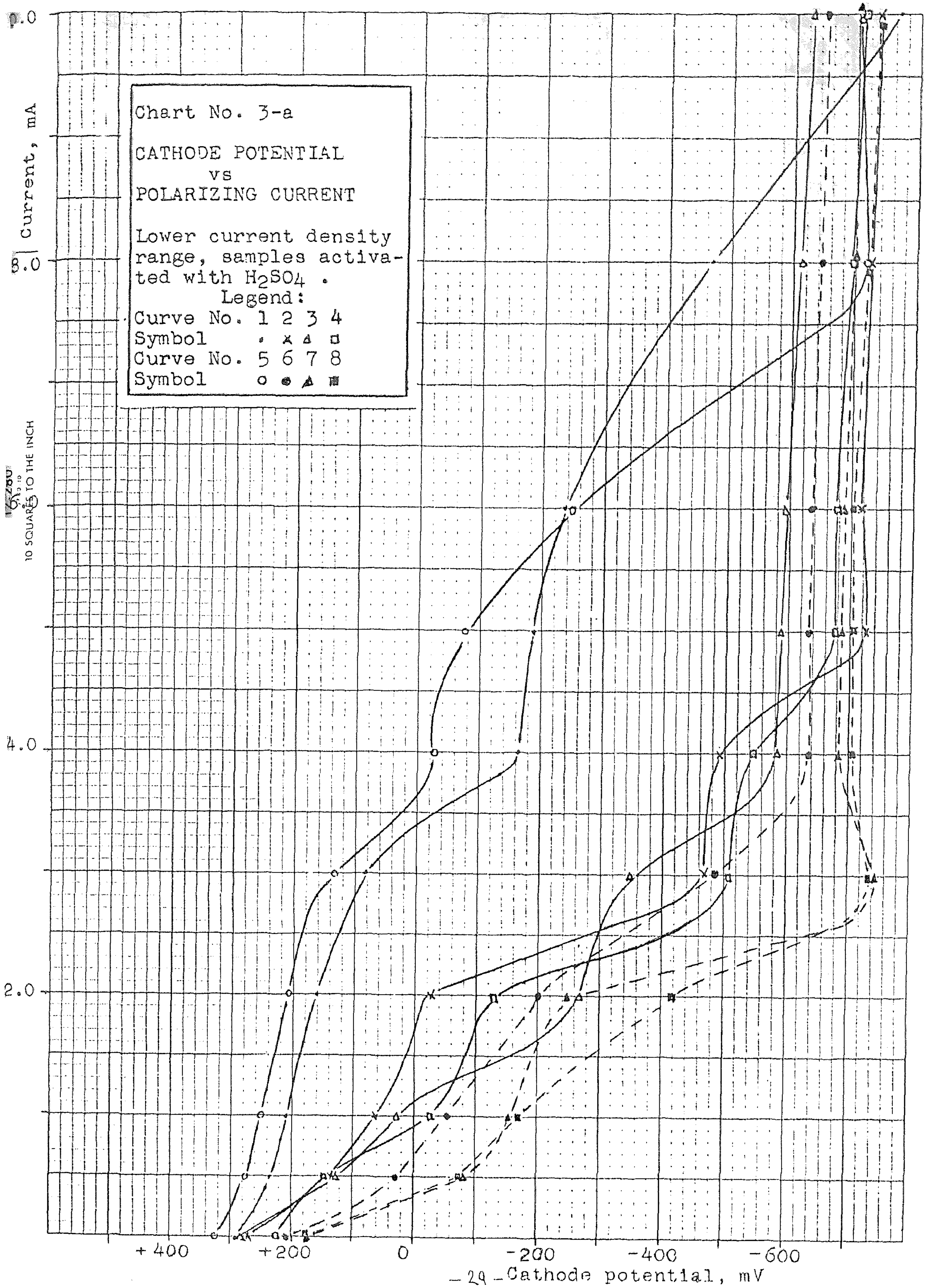


Chart No. 3-a
CATHODE POTENTIAL
vs
POLARIZING CURRENT
Lower current density range, samples activated with $H_2SO_4$.
Legend:
Curve No. 1 2 3 4
Symbol · x △ □
Curve No. 5 6 7 8
Symbol ○ ● ▲ ■
Current, mA
10.0
8.0
6.0
4.0
2.0
+400
+200
0
-200
-400
-600
Cathode potential, mV
10 SQUARES TO THE INCH

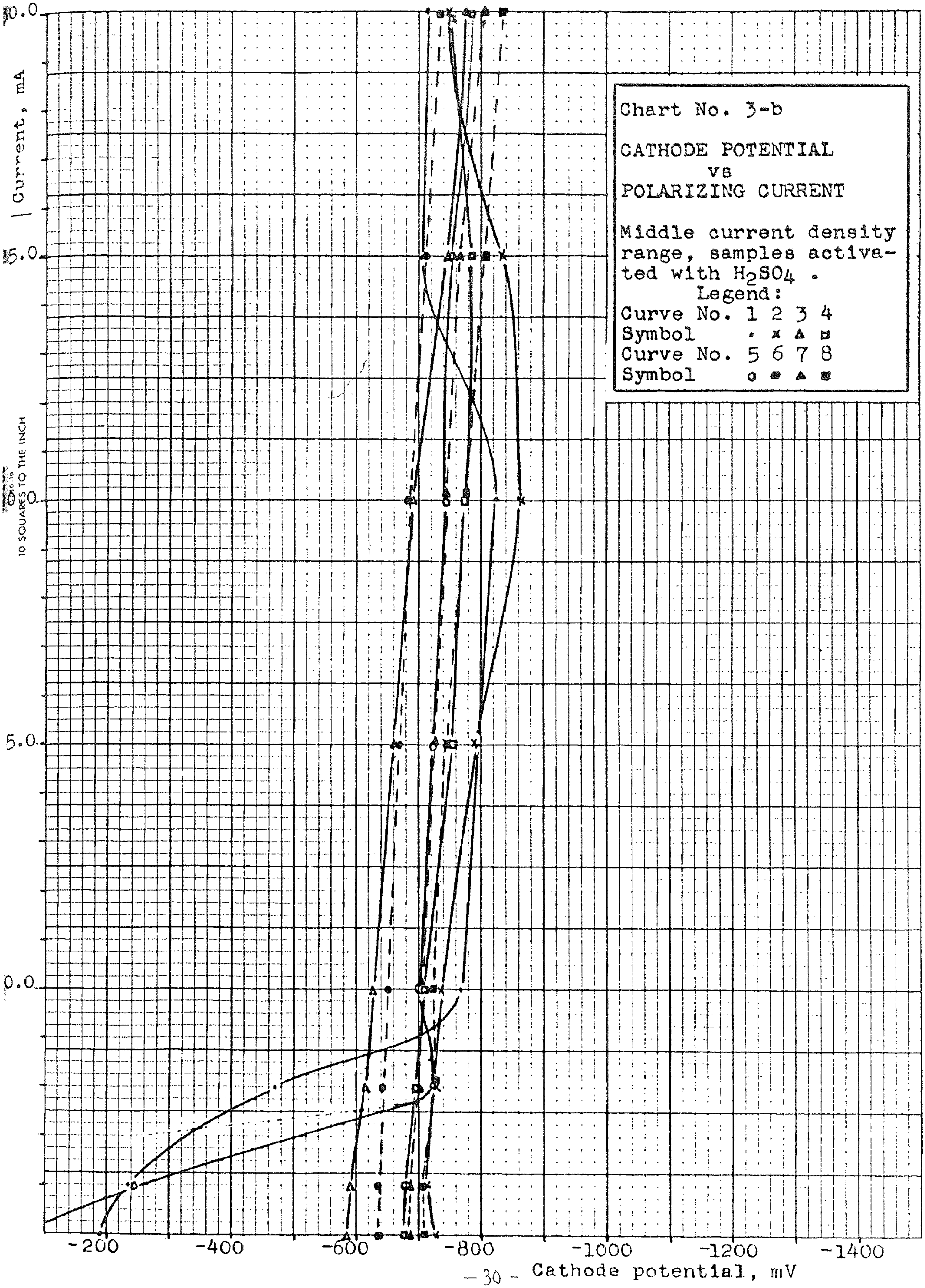


Chart No. 3-b
CATHODE POTENTIAL
vs
POLARIZING CURRENT
Middle current density range, samples activated with $H_2SO_4$ .
Legend:
Curve No. 1 2 3 4
Symbol · × △ ◻
Curve No. 5 6 7 8
Symbol ○ ● ▲ ■
Current, mA
0.0
5.0
0
5.0
0.0
10 SQUARES TO THE INCH
-200
-400
-600
-800
-1000
-1200
-1400
Cathode potential, mV

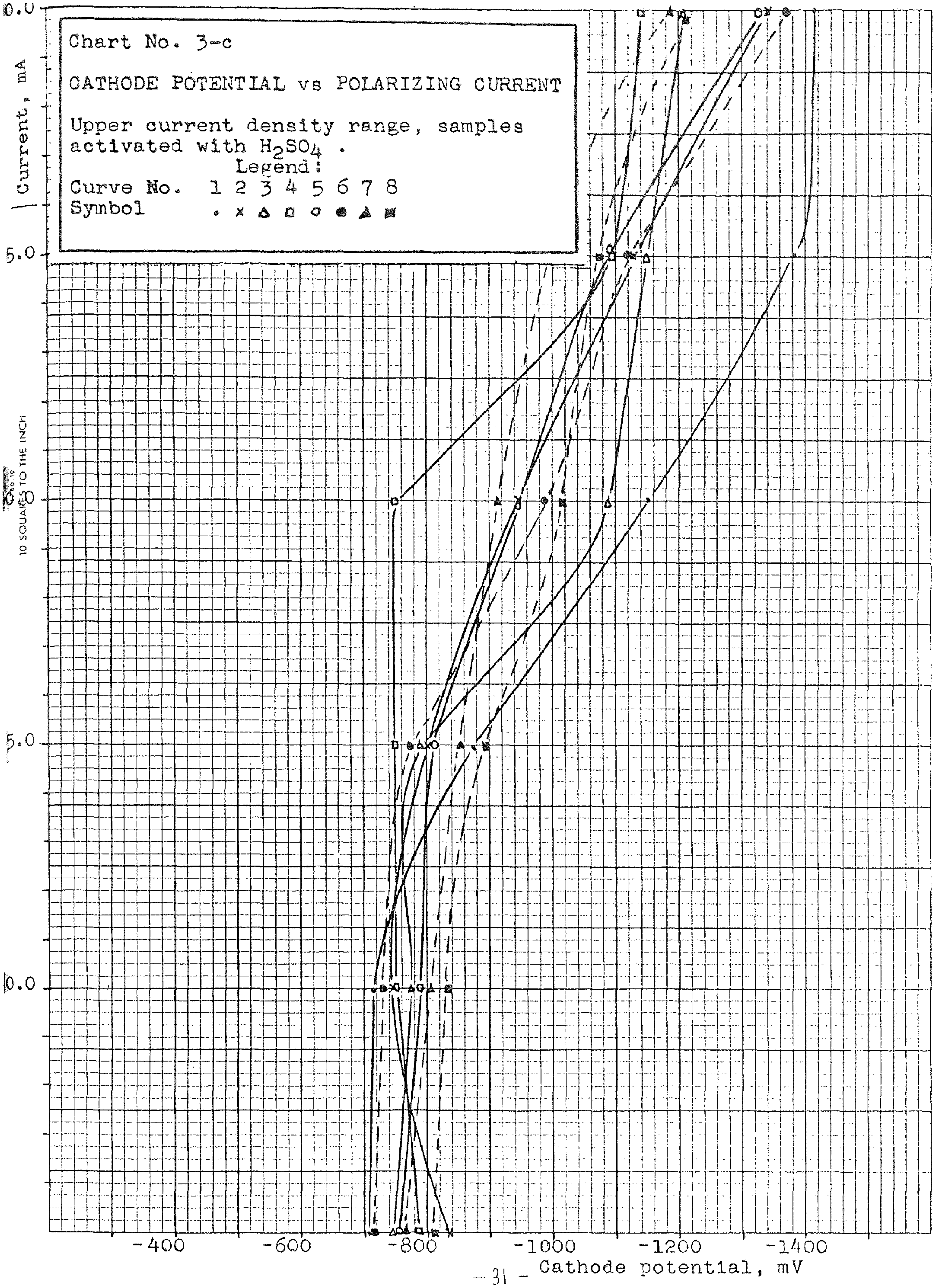


Chart No. 3-c

CATHODE POTENTIAL vs POLARIZING CURRENT

Upper current density range, samples activated with $H_2SO_4$ .

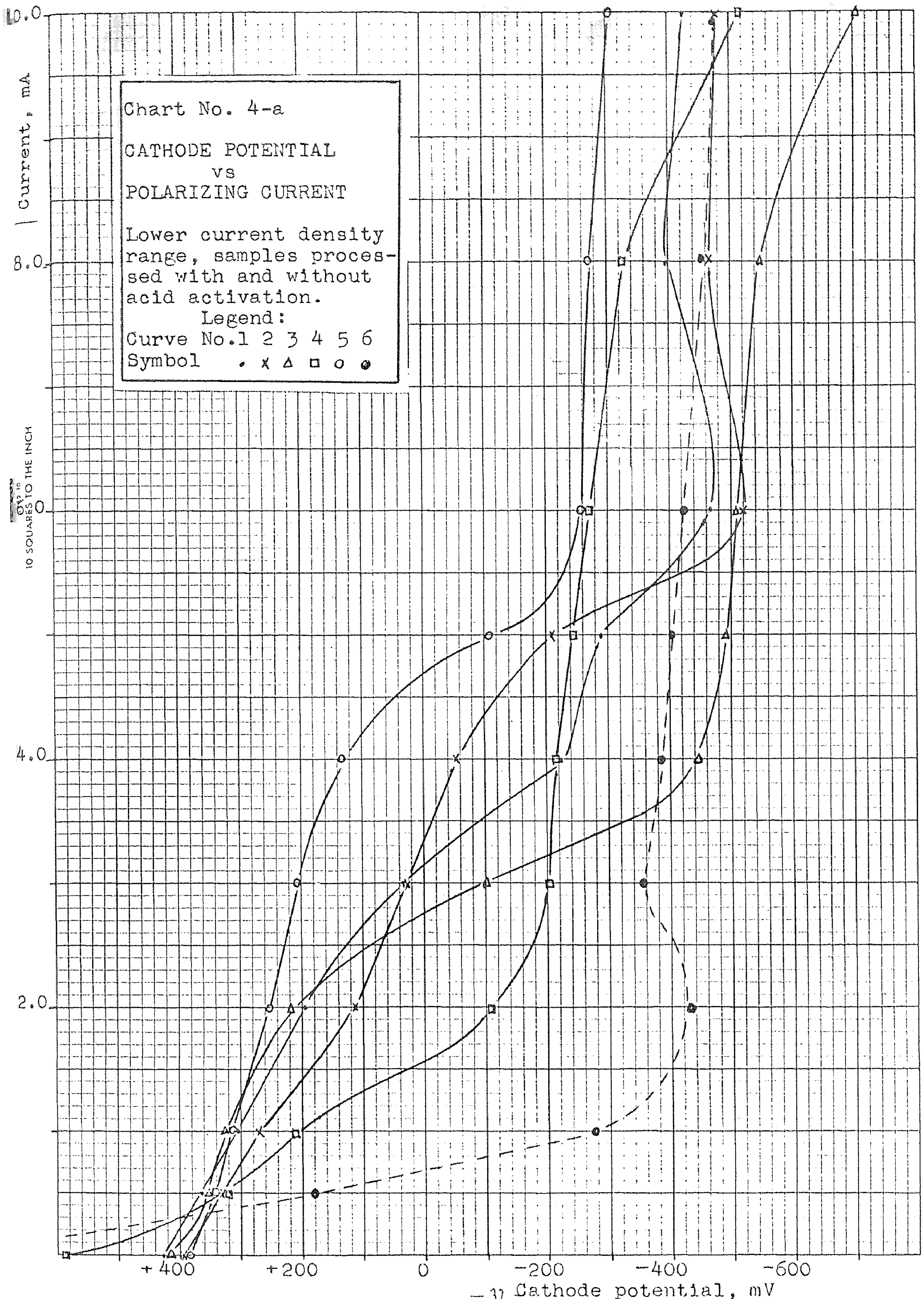
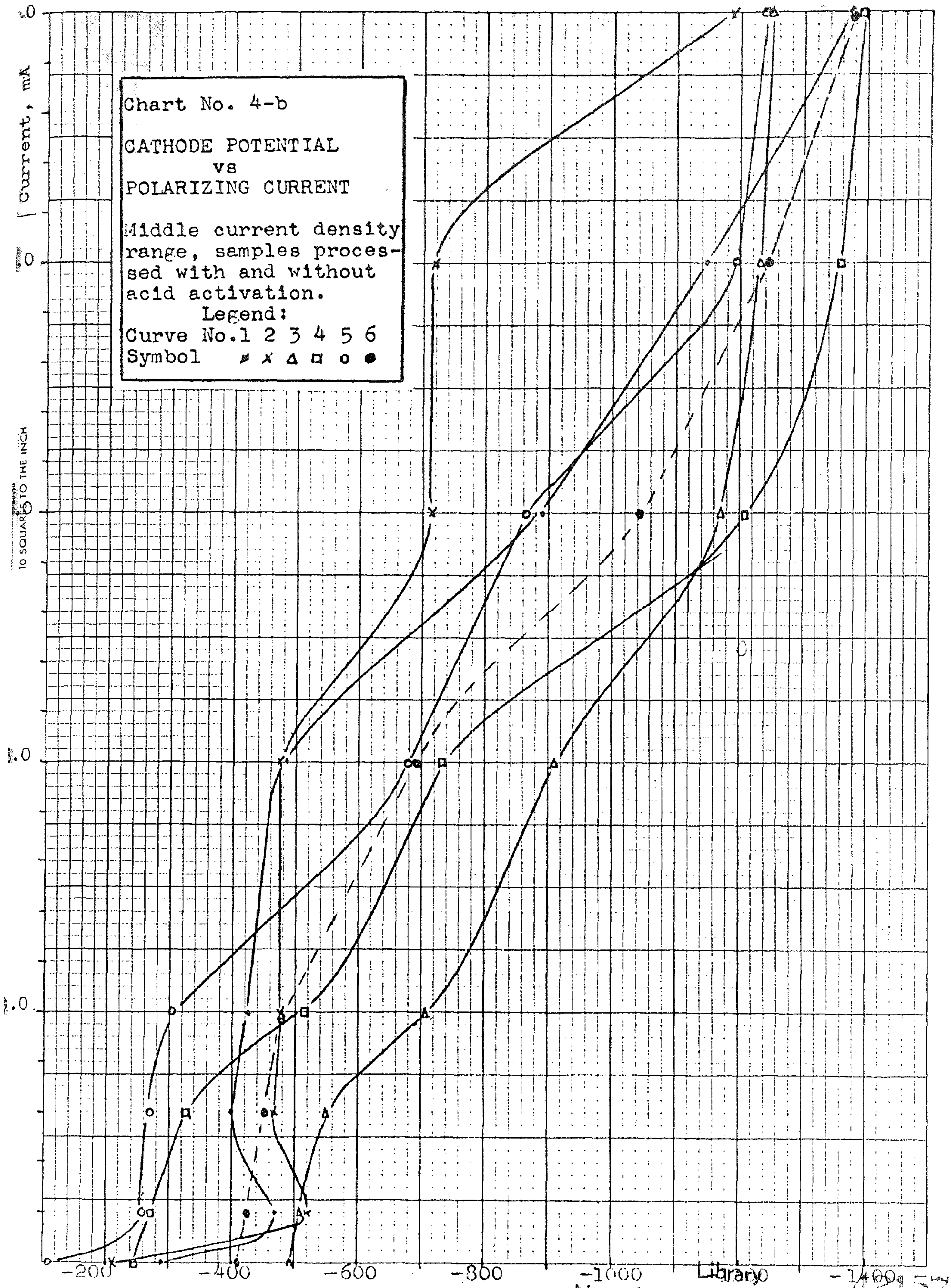


Chart No. 4-a
CATHODE POTENTIAL
vs
POLARIZING CURRENT
Lower current density range, samples processed with and without acid activation.
Legend:
Curve No. 1 2 3 4 5 6
Symbol • x △ □ ○ ●
Current, mA
10.0
8.0
6.0
4.0
2.0
+400
+200
0
-200
-400
-600
Cathode potential, mV
10 SQUARES TO THE INCH



Cathode Potential, mV

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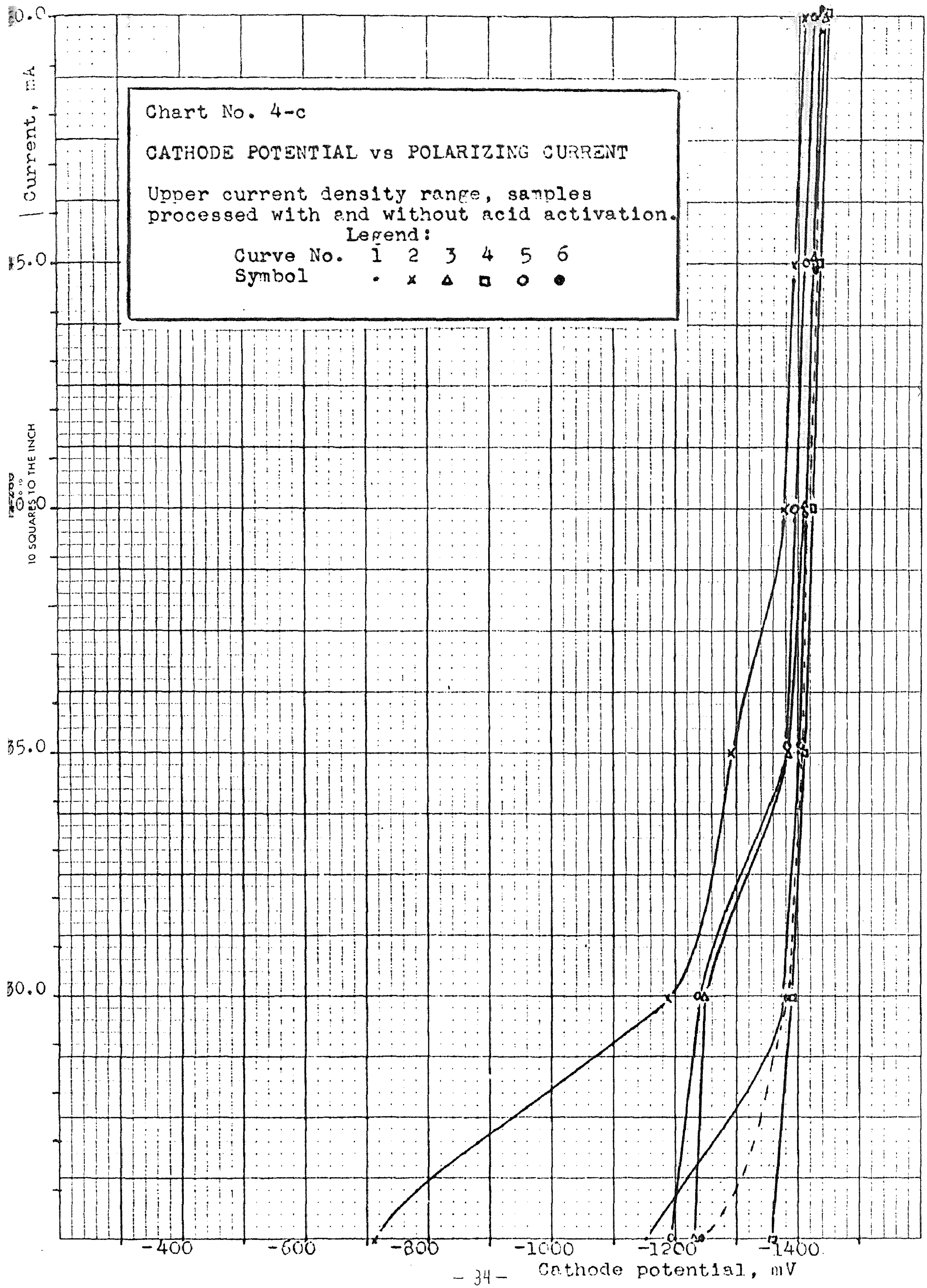

Chart No. 4-c

CATHODE POTENTIAL vs POLARIZING CURRENT

Upper current density range, samples processed with and without acid activation.

Legend:

| Curve No. | 1 | 2 | 3 | 4 | 5 | 6 |
|---|---|---|---|---|---|---|
| Symbol | • | x | △ | □ | ○ | ● |

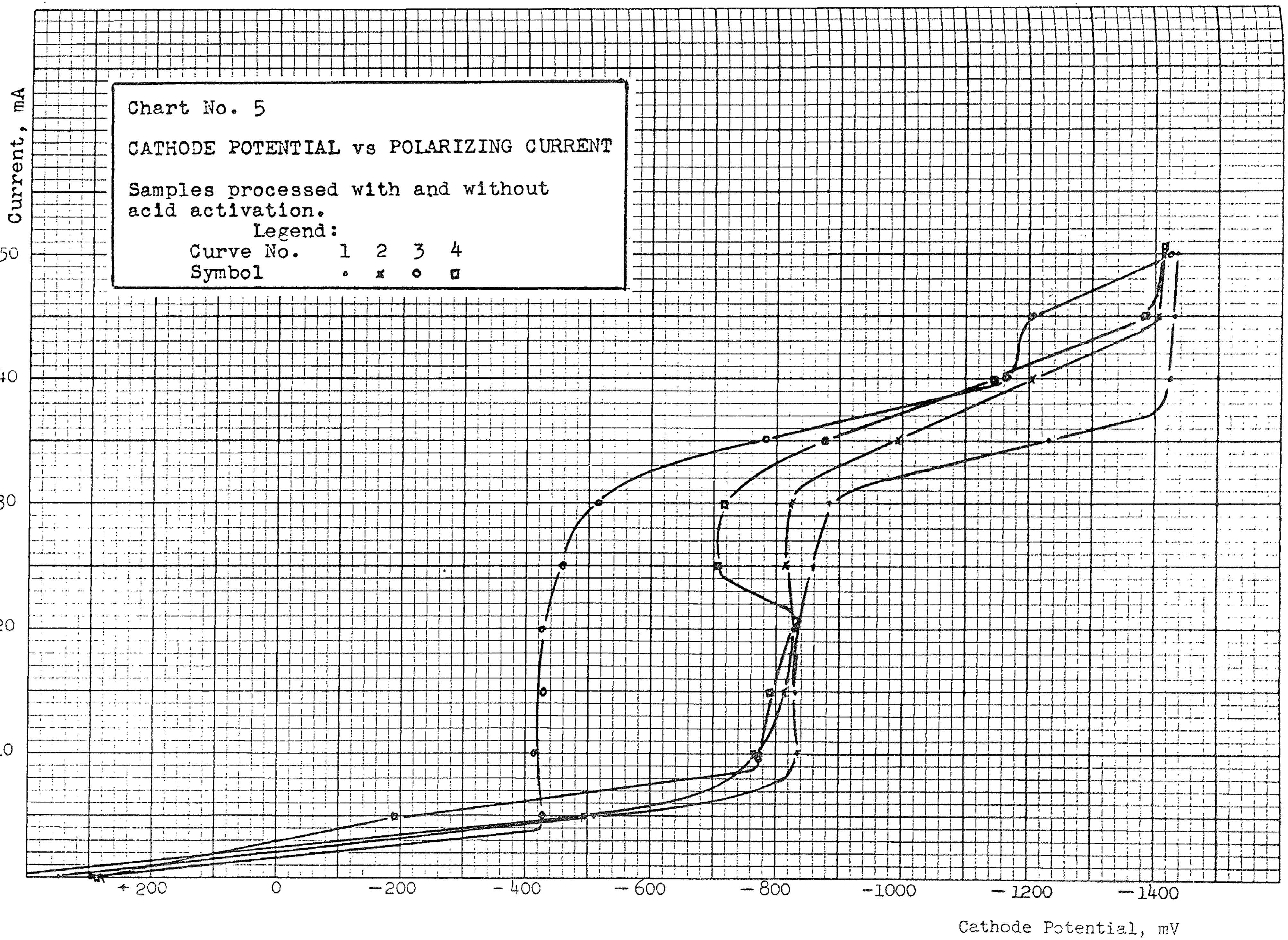

Chart No. 5

CATHODE POTENTIAL vs POLARIZING CURRENT

Samples processed with and without acid activation.

10 SQUARES TO THE INCH

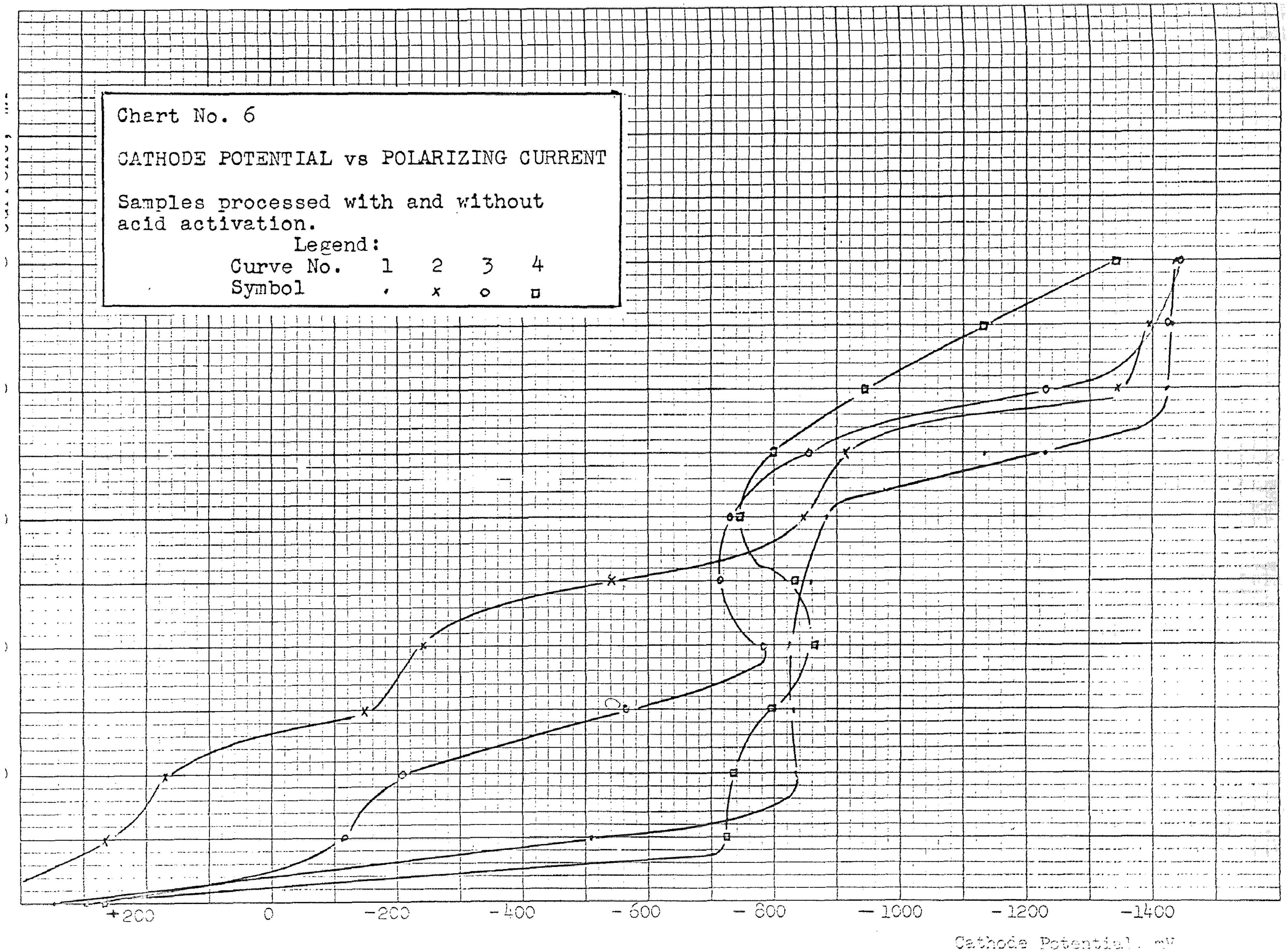

Chart No. 6

CATHODE POTENTIAL vs POLARIZING CURRENT

Samples processed with and without acid activation.

Legend:

| Curve No. | 1 | 2 | 3 | 4 |
|---|---|---|---|---|
| Symbol | • | x | o | □ |

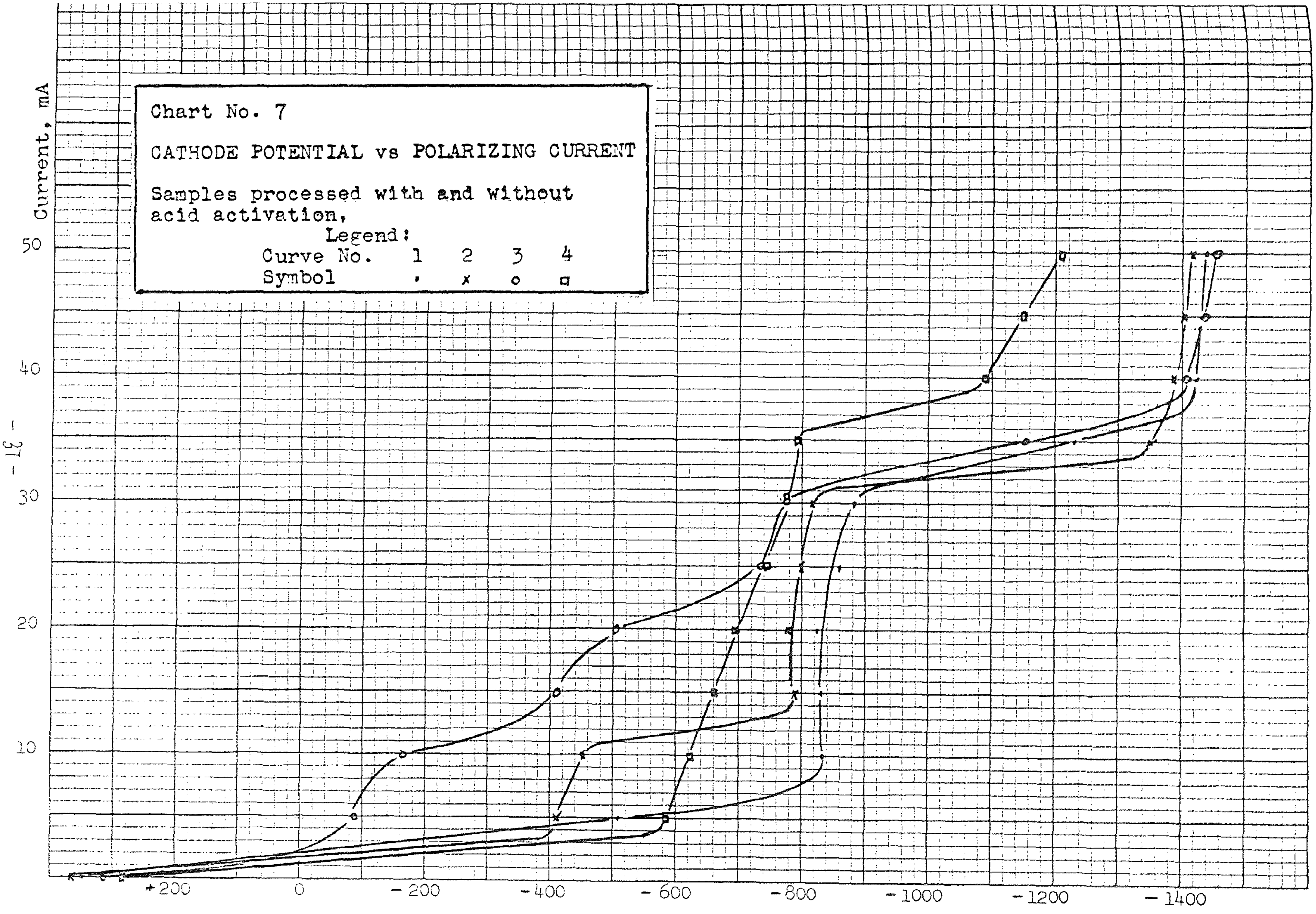

Chart No. 7

CATHODE POTENTIAL vs POLARIZING CURRENT

Samples processed with and without acid activation.

Legend:

| Curve No. | 1 | 2 | 3 | 4 |
|---|---|---|---|---|
| Symbol | • | x | o | ◻ |

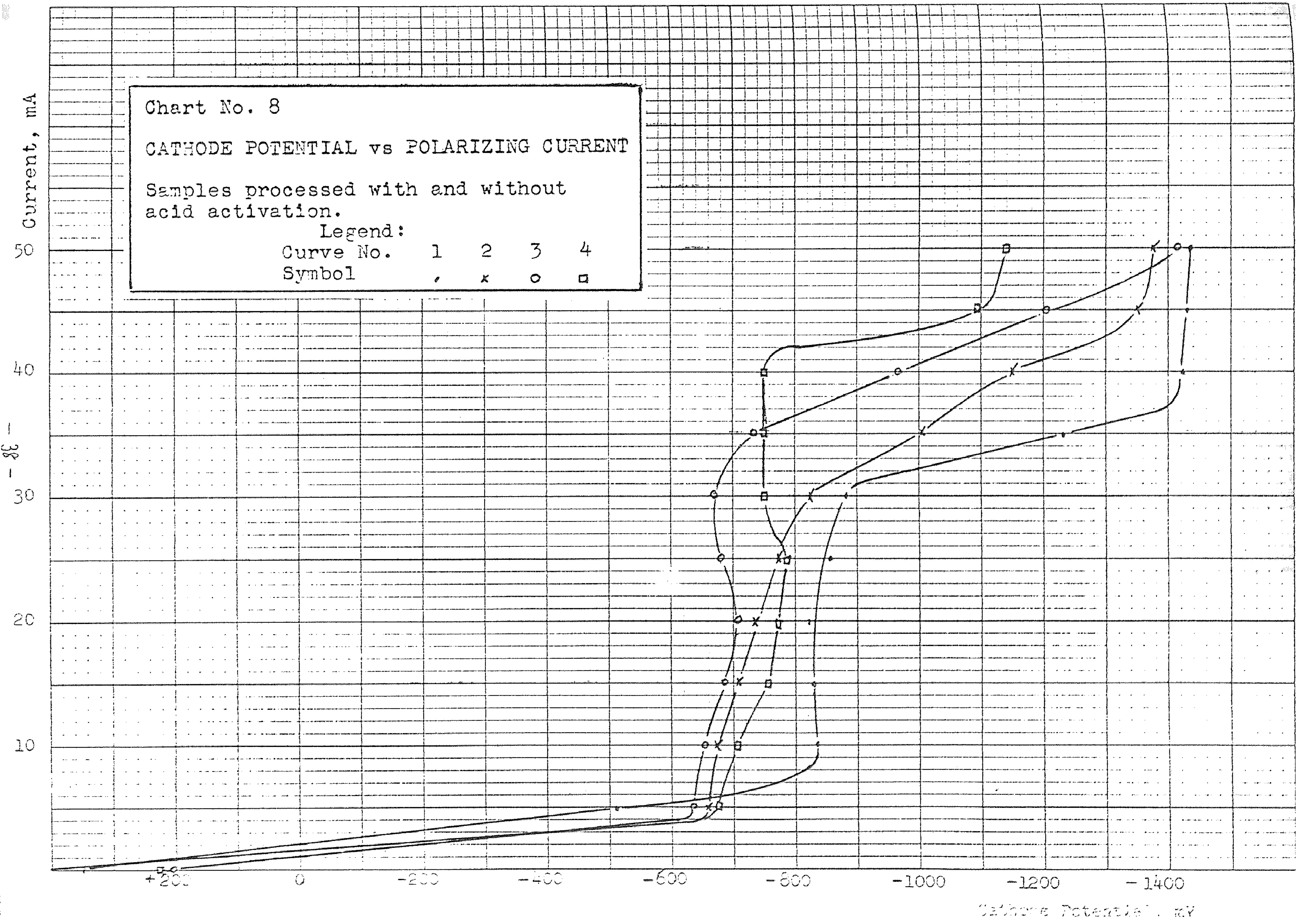

Chart No. 8

CATHODE POTENTIAL vs POLARIZING CURRENT

Samples processed with and without acid activation.

Legend:

| Curve No. | 1 | 2 | 3 | 4 |
|---|---|---|---|---|
| Symbol | , | x | o | □ |

10 SQUARES TO THE INCH

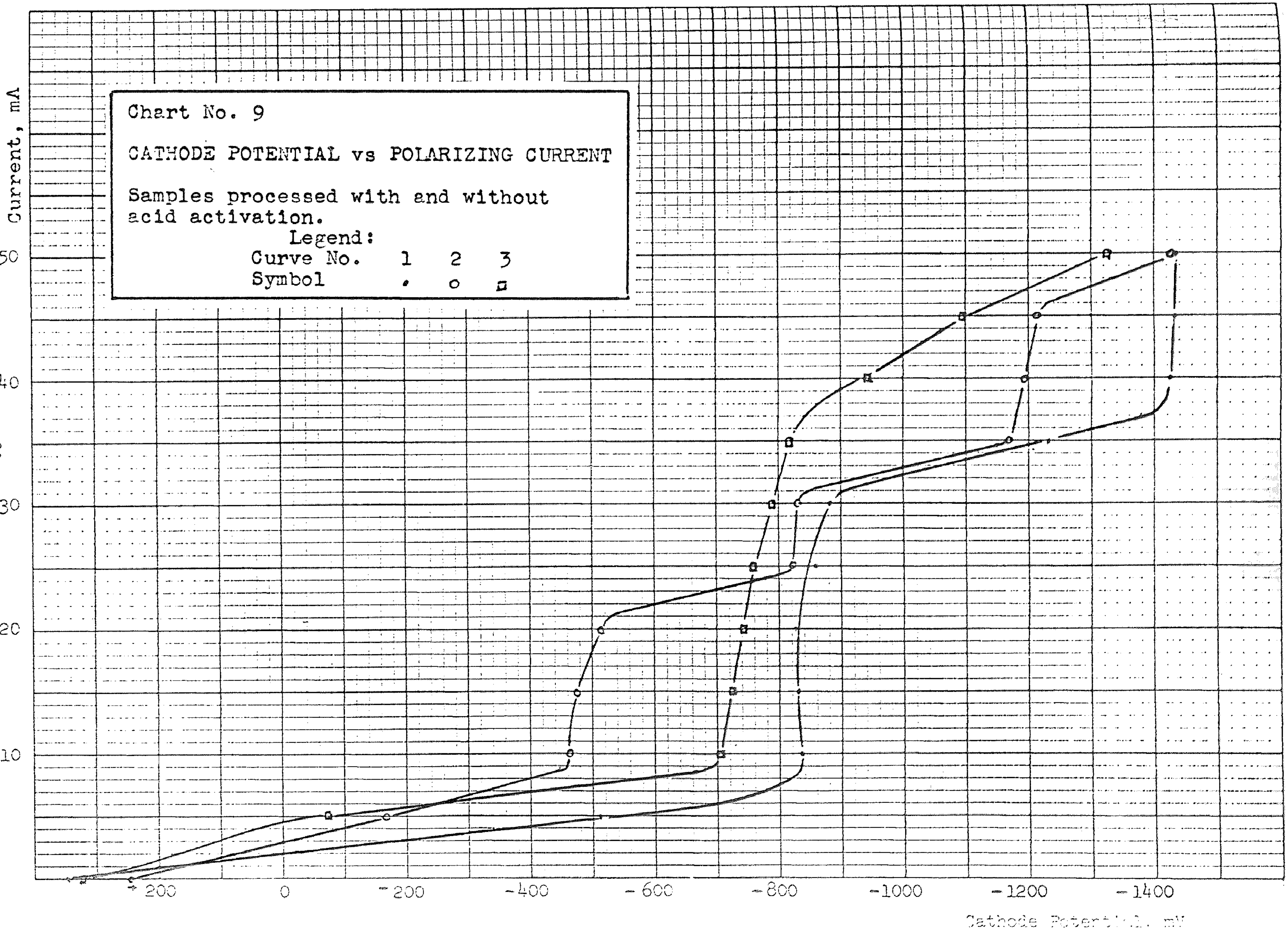

Chart No. 9

CATHODE POTENTIAL vs POLARIZING CURRENT

Samples processed with and without acid activation.

Legend:

| Curve No. | 1 | 2 | 3 |
|---|---|---|---|
| Symbol | • | ○ | ◻ |

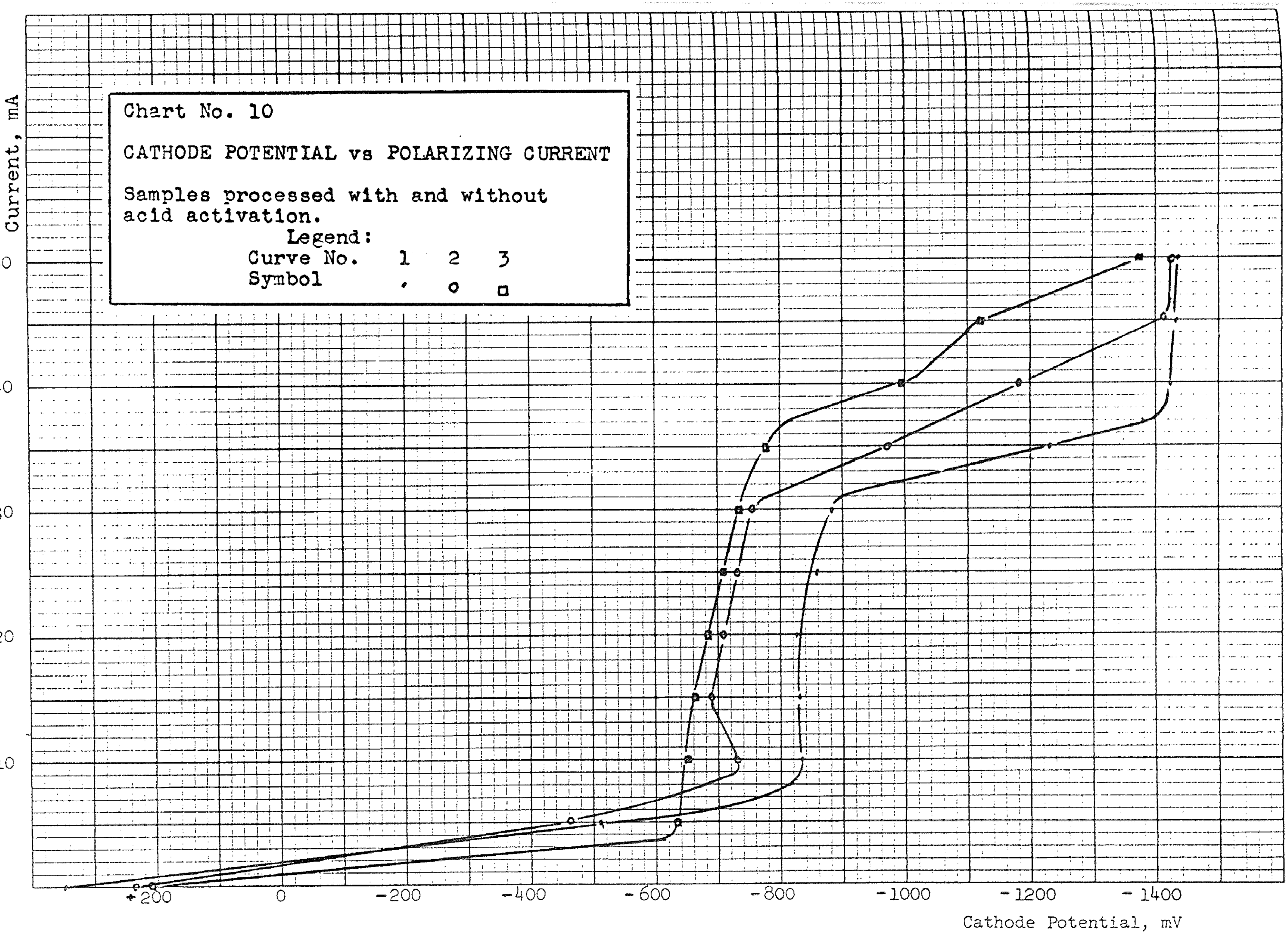

Chart No. 10

CATHODE POTENTIAL vs POLARIZING CURRENT

Samples processed with and without acid activation.

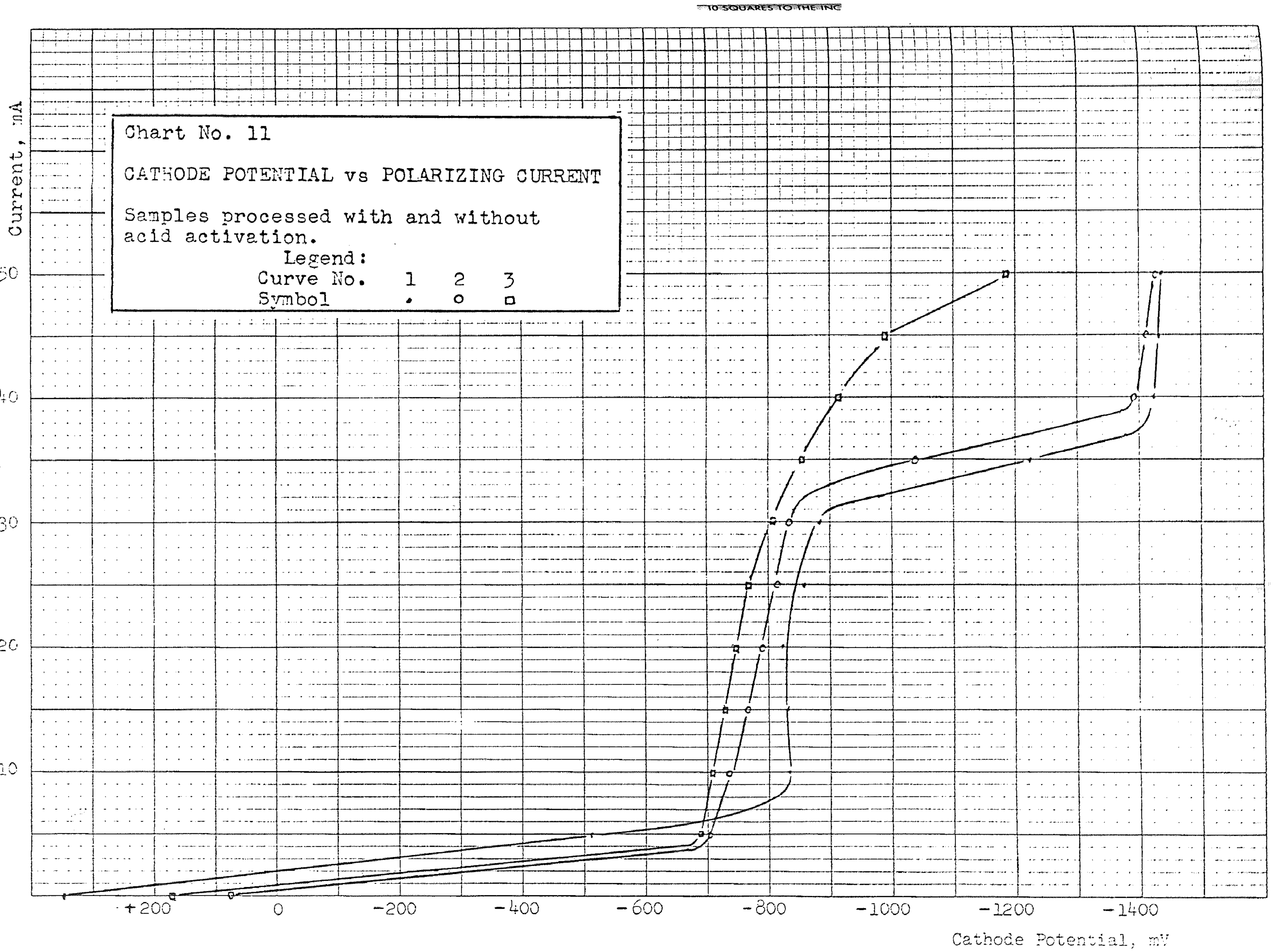

Chart No. 11

CATHODE POTENTIAL vs POLARIZING CURRENT

Samples processed with and without acid activation.

Legend:

| Curve No. | 1 | 2 | 3 |
|---|---|---|---|
| Symbol | • | ○ | □ |

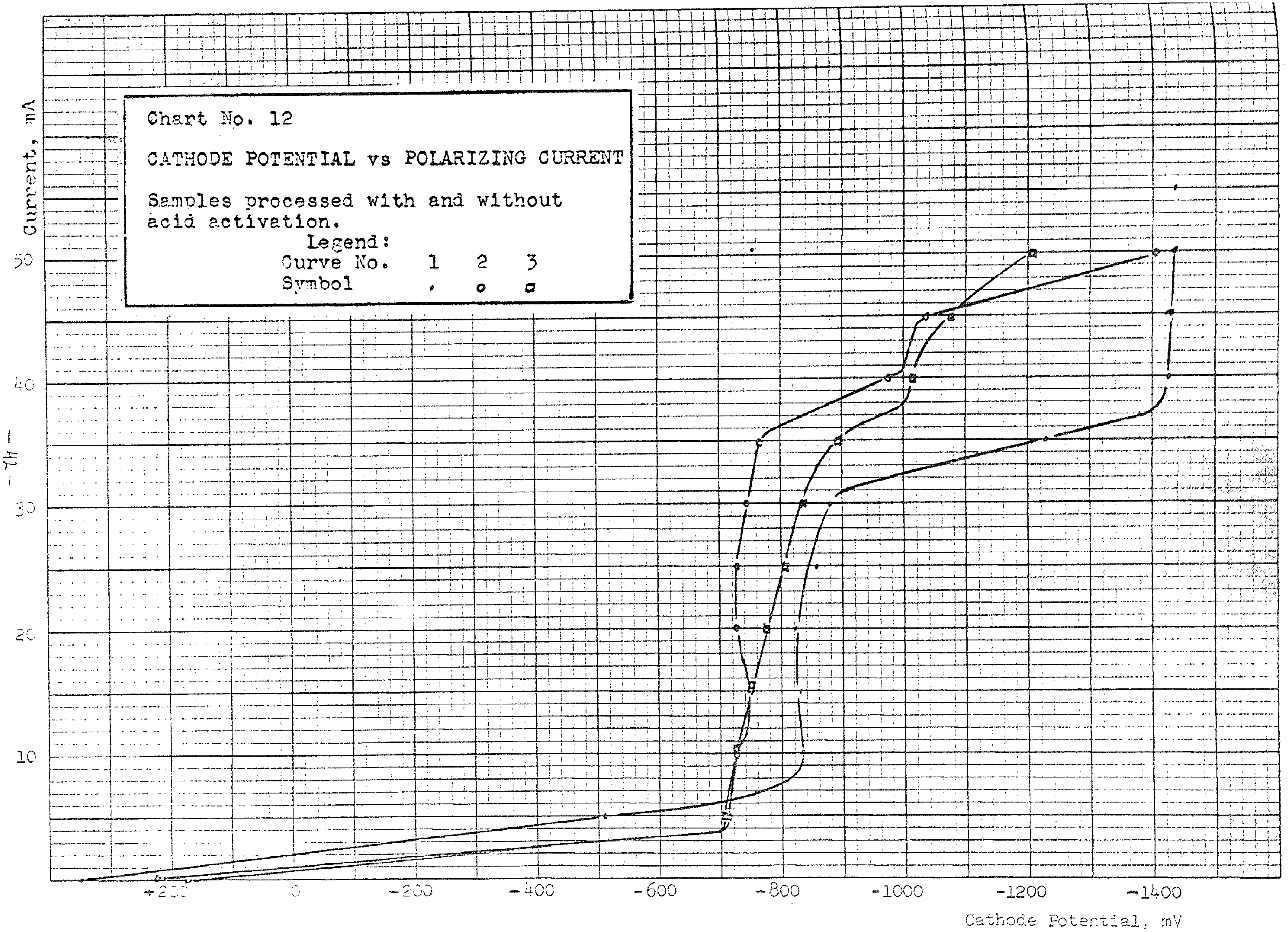

Chart No. 12

CATHODE POTENTIAL vs POLARIZING CURRENT

Samples processed with and without acid activation.

# GRAPHICAL DATA LOCATOR

| Curve No. | Symbol | Chart No. | Data in Table No. | Cleaning cycle No. | Cleaning Cycle Synopsis |
|---|---|---|---|---|---|
| 1 | · | 1a,b.c | 1 | 1 | Sanded, degreased |
| 2 | × | 1a,b,c | 2 | 2 | Sanded, degreased, wet scoured |
| 3 | △ | 1a,b,c | 3 | 3 | Sanded, degreased, soak cleaned |
| 4 | □ | 1a,b,c | 4 | 4 | Sanded, degreased, cathodic. cleaned |
| 5 | ○ | 1a,b,c | 5 | 5 | Sanded, degreased, anodic. cleaned |
| 1 | · | 2a,b,c | 6 | 6 | Sanded, degreased, HCl pickled |
| 2 | × | 2a,b,c | 7 | 7 | Sanded, degreased, soak cl.,HCl pickled |
| 3 | △ | 2a,b,c | 8 | 8 | Sanded, degreased cath. cl.,HCl pickled |
| 4 | □ | 2a,b,c | 9 | 9 | Sanded, degreased, anod.cl.,HCl pickled |
| 5 | ○ | 2a,b,c | 14 | 14 | Sand.,deg.,cath.cl., HCl, cath.cl., HCl |
| 6 | ● | 2a,b,c | 15 | 15 | Sand., deg., anod.cl., HCl, cath.cl., HCl |
| 7 | ▲ | 2a,b,c | 16 | 16 | Sand., deg., cath.cl., HCl, anod. cl., HCl |
| 8 | ■ | 2a,b,c | 17 | 17 | Sand., deg., anod. cl., HCl, anod. cl., HCl |
| 1 | · | 3a,b,c | 10 | 10 | Sanded, degreased, $H_2SO_4$ pickled |
| 2 | × | 3a,b,c | 11 | 11 | Sanded, degreased, soak cl., $H_2SO_4$ pickled |
| 3 | △ | 3a,b,c | 12 | 12 | Sanded, degreased, cath. cl., $H_2SO_4$ |
| 4 | □ | 3a,b,c | 13 | 13 | Sanded, degreased, anod. cl., $H_2SO_4$ |

| Curve No. | Symbol | Chart No. | Data in Table No. | Cleaning cycle No. | Cleaning Cycle Synopsis |
|---|---|---|---|---|---|
| 5 | ○ | 3a,b,c | 18 | 18 | Sand.,deg.,cath.cl., [illegible] HCl, cath.,$H_2SO_4$ |
| 6 | ● | 3a,b,c | 19 | 19 | Sand.,deg.,anod.cl, HCl,cath.cl.,$H_2SO_4$ |
| 7 | ▲ | 3a,b,c | 20 | 20 | Sand.,deg.,cath.cl., HCl,anod.cl.,$H_2SO_4$ |
| 8 | ▣ | 3a,b,c | 21 | 21 | Sand.,deg.,anod.cl, HCl,anod.cl.,$H_2SO_4$ |
| 1 | • | 4a,b,c | 22 | 7 | Sanded,degreased, soak cl.,HCl pickled |
| 2 | × | 4a,b,c | 23 | 11 | Sanded, degreased, soak cl.,$H_2SO_4$ pickl. |
| 3 | △ | 4a,b,c | 24 | 1 | Sanded, degreased |
| 4 | □ | 4a,b,c | 25 | 3 | Sanded, degreased, soak cleaned |
| 5 | ○ | 4a,b,c | 26 | 4 | Sanded, degreased, cathodic. cleaned |
| 6 | ● | 4a,b,c | 27 | 5 | Sanded, degreased, anodic. cleaned |
| 1 | • | 5 | 2 | 2 | Sanded, degreased, wet scoured |
| 2 | × | 5 | 1 | 1 | Sanded, degreased |
| 3 | ○ | 5 | 6 | 6 | Sanded, degreased, HCl pickled |
| 4 | □ | 5 | 10 | 10 | Sanded, degreased, $H_2SO_4$ pickled |
| 1 | • | 6 | 2 | 2 | Sanded, degreased, wet scoured |
| 2 | × | 6 | 3 | 3 | Sanded, degreased, soak cleaned |
| 3 | ○ | 6 | 7 | 7 | Sanded, degreased, soak cl.,HCl picled |
| 4 | □ | 6 | 11 | 11 | Sanded, degreased, soak cl.,$H_2SO_4$ pickl. |

| Curve No. | Symbol | Chart No. | Data in Table No. | Cleaning Cycle No. | Cleaning Cycle Synopsis |
|---|---|---|---|---|---|
| 1 | • | 7 | 2 | 2 | Sanded, degreased, wet scoured |
| 2 | × | 7 | 4 | 4 | Sanded, degreased, cathodic. cleaned |
| 3 | ○ | 7 | 8 | 8 | Sanded, degreased, cath.cl.,HCl pickled |
| 4 | □ | 7 | 12 | 12 | Sanded, degreased, cath.cl.,$H_2SO_4$ pickl. |
| 1 | • | 8 | 2 | 2 | Sanded, degreased, wet scoured |
| 2 | × | 8 | 5 | 5 | Sanded, degreased, anodic. cleaned |
| 3 | ○ | 8 | 9 | 9 | Sanded, degreased, anod.cl.,HCl pickl. |
| 4 | □ | 8 | 13 | 13 | Sanded, degreased, anod.cl.,$H_2SO_4$ pickl. |
| 1 | • | 9 | 2 | 2 | Sanded, degreased, wet scoured |
| 2 | ○ | 9 | 14 | 14 | Sand.,deg.,cath.cl., HCl,cath.cl.,HCl |
| 3 | □ | 9 | 18 | 18 | Sand.,deg.,cath.cl., HCl,cath.cl.,$H_2SO_4$ |
| 1 | • | 10 | 2 | 2 | Sanded, degreased, wet scoured |
| 2 | ○ | 10 | 15 | 15 | Sand.,deg.,anod.cl., HCl,cath.cl.,HCl |
| 3 | □ | 10 | 19 | 19 | Sand.,deg.,anod.cl., HCl.cath.cl.,$H_2SO_4$ |
| 1 | • | 11 | 2 | 2 | Sanded, degreased, wet scoured |
| 2 | ○ | 11 | 16 | 16 | Sand.,deg.,cath.cl., HCl,anod.cl.,HCl |
| 3 | □ | 11 | 20 | 20 | Sand.,deg.,cath.cl., HCl,anod.cl.,$H_2SO_4$ |
| 1 | • | 12 | 2 | 2 | Sanded,degreased, wet scoured |
| 2 | ○ | 12 | 17 | 17 | Sand.,deg.,anod.cl., HCl,anod.cl.,HCl |
| 3 | □ | 12 | 21 | 21 | sand.,deg.,anod.cl., HCl,anod.cl.,$H_2SO_4$ |

TABLE No.1

Data for Curve No.1 on Charts No. 1a, 1b, 1c,
and for Curve No.2 on Chart No.5 .
Sample preparation: cleaning cycle No.1
Apparent cathode area: 0.114 sq.in.

| Polariz. current, mA | Cathode current density mA/sq.in. | Cathode Potential, mV | | | |
|---|---|---|---|---|---|
| | | 1 | 2 | 3 | Average |
| 0.0 | 0.0 | + 445 | + 465 | + 425 | + 445 |
| 0.5 | 4.4 | + 305 | + 315 | + 295 | + 305 |
| 1.0 | 8.8 | + 280 | + 295 | + 245 | + 273 |
| 2.0 | 17.6 | + 155 | + 260 | + 30 | + 115 |
| 3.0 | 26.4 | + 5 | + 155 | - 320 | - 53 |
| 4.0 | 35.2 | - 385 | - 520 | - 820 | - 575 |
| 5.0 | 44.0 | - 725 | - 15 | - 745 | - 495 |
| 6.0 | 52.8 | - 735 | - 735 | - 745 | - 738 |
| 8.0 | 70.4 | - 750 | - 845 | - 765 | - 787 |
| 10.0 | 88.0 | - 770 | - 745 | - 775 | - 763 |
| 15.0 | 132.0 | - 830 | - 795 | - 820 | - 815 |
| 20.0 | 176.0 | - 870 | - 805 | - 835 | - 837 |
| 25.0 | 220.0 | - 745 | - 845 | - 855 | - 815 |
| 30.0 | 264.0 | - 780 | - 845 | - 845 | - 823 |
| 35.0 | 308.0 | - 870 | - 805 | -1310 | - 995 |
| 40.0 | 352.0 | -1420 | - 845 | -1355 | -1205 |
| 45.0 | 396.0 | -1435 | -1385 | -1385 | -1402 |
| 50.0 | 440.0 | -1445 | -1405 | -1395 | -1415 |

TABLE No.2

Data for Curve No.2 on Charts No. 1a, 1b, 1c,
and for Curve No.1 on Charts No.5 to No.12
Sample preparation: cleaning cycle No.2.
Apparent cathode area: 0.114 sq.in.

| Polariz. current, mA | Cathode current density, mA/sq.in | Cathode Potential, mV | | | |
|---|---|---|---|---|---|
| | | 1 | 2 | 3 | Average |
| 0.0 | 0.0 | + 335 | + 360 | + 345 | + 347 |
| 0.5 | 4.4 | + 240 | + 260 | + 245 | + 248 |
| 1.0 | 8.8 | + 210 | + 240 | + 180 | + 210 |
| 2.0 | 17.6 | - 40 | + 160 | + 10 | + 43 |
| 3.0 | 26.4 | - 645 | + 95 | - 855 | - 468 |
| 4.0 | 35.2 | - 685 | + 25 | - 745 | - 468 |
| 5.0 | 44.0 | - 710 | - 70 | - 755 | - 512 |
| 6.0 | 52.8 | - 735 | - 820 | - 755 | - 770 |
| 8.0 | 70.4 | - 755 | - 870 | - 755 | - 795 |
| 10.0 | 88.0 | - 870 | - 885 | - 755 | - 837 |
| 15.0 | 132.0 | - 805 | - 895 | - 795 | - 832 |
| 20.0 | 176.0 | - 845 | - 820 | - 810 | - 825 |
| 25.0 | 220.0 | - 895 | - 845 | - 840 | - 860 |
| 30.0 | 264.0 | - 920 | - 870 | - 855 | - 882 |
| 35.0 | 308.0 | -1405 | -1395 | - 895 | -1232 |
| 40.0 | 352.0 | -1430 | -1430 | -1405 | -1422 |
| 45.0 | 396.0 | -1435 | -1435 | -1430 | -1433 |
| 50.0 | 440.0 | -1440 | -1440 | -1435 | -1438 |

TABLE No.3

Data for Curve No.3 on Charts No. 1a, 1b, 1c,
and for Curve No.2 on Chart No.6 .
Sample preparation: cleaning cycle No.3 .
Apparent cathode area: 0.114 sq.in.

| Polariz. current, mA | Cathode current density mA/sq.in | Cathode Potential, mV | | | |
|---|---|---|---|---|---|
| | | 1 | 2 | 3 | Average |
| 0.0 | 0.0 | + 455 | + 465 | + 455 | + 458 |
| 0.5 | 4.4 | + 330 | + 340 | + 340 | + 370 |
| 1.0 | 8.8 | + 320 | + 340 | + 320 | + 327 |
| 2.0 | 17.6 | + 305 | + 315 | + 310 | + 310 |
| 3.0 | 26.4 | + 290 | + 305 | + 300 | + 298 |
| 4.0 | 35.2 | + 270 | + 280 | + 290 | + 280 |
| 5.0 | 44.0 | + 265 | + 265 | + 270 | + 267 |
| 6.0 | 52.8 | + 255 | + 230 | + 260 | + 248 |
| 8.0 | 70.4 | + 225 | + 140 | + 245 | + 203 |
| 10.0 | 88.0 | + 210 | + 70 | + 230 | + 170 |
| 15.0 | 132.0 | + 145 | - 830 | + 245 | - 147 |
| 20.0 | 176.0 | - 30 | - 820 | +130 | - 240 |
| 25.0 | 220.0 | - 855 | - 820 | + 55 | - 540 |
| 30.0 | 264.0 | - 850 | - 835 | - 855 | - 845 |
| 35.0 | 308.0 | - 945 | - 895 | - 895 | - 912 |
| 40.0 | 352.0 | -1400 | -1290 | -1335 | -1343 |
| 45.0 | 396.0 | -1430 | -1370 | -1385 | -1395 |
| 50.0 | 440.0 | - - | - | - | - |

TABLE No.4

Data for Curve No.4 on Charts No. 1a, 1b, 1c,
and for Curve No.2 on Chart No.7 .
Sample preparation: cleaning cycle No.4 .
Apparent cathode area: 0.114 sq.in.

| Polariz. current, mA | Cathode current density mA/sq.in | Cathode Potential, mV | | | |
|---|---|---|---|---|---|
| | | 1 | 2 | 3 | Average |
| 0.0 | 0.0 | + 355 | + 390 | + 360 | + 368 |
| 0.5 | 4.4 | + 280 | + 305 | + 280 | + 322 |
| 1.0 | 8.8 | + 230 | + 285 | + 230 | + 248 |
| 2.0 | 17.6 | + 130 | + 265 | + 110 | + 168 |
| 3.0 | 26.4 | + 40 | + 250 | - 35 | + 85 |
| 4.0 | 35.2 | - 80 | + 230 | - 870 | - 240 |
| 5.0 | 44.0 | - 720 | + 215 | - 745 | - 417 |
| 6.0 | 52.8 | - 645 | + 190 | - 730 | - 396 |
| 8.0 | 70.4 | - 655 | + 155 | - 745 | - 415 |
| 10.0 | 88.0 | - 685 | + 65 | - 750 | - 457 |
| 15.0 | 132.0 | - 740 | - 845 | - 790 | - 790 |
| 20.0 | 176.0 | - 770 | - 775 | - 795 | - 780 |
| 25.0 | 220.0 | - 795 | - 795 | - 805 | - 798 |
| 30.0 | 264.0 | - 820 | - 815 | - 820 | - 818 |
| 35.0 | 308.0 | -1345 | -1375 | -1330 | -1350 |
| 40.0 | 352.0 | -1380 | -1405 | -1360 | -1382 |
| 45.0 | 396.0 | -1400 | -1425 | -1380 | -1402 |
| 50.0 | 440.0 | - | -1435 | -1395 | -1415 |

TABLE No.5

Data for Curve No.5 on Chart No. 1a, 1b, 1c,
and for Curve No.2 on Chart No.8 .
Sample preparation: cleaning cycle No.5 .
Apparent cathode area: 0.114 sq.in.

| Polariz. current, mA | Cathode current density mA/sq.in | Cathode Potential, mV |  |  |  |
|---|---|---|---|---|---|
|  |  | 1 | 2 | 3 | Average |
| 0.0 | 0.0 | +465 | + 445 | + 460 | + 457 |
| 0.5 | 4.4 | + 295 | + 280 | + 265 | + 280 |
| 1.0 | 8.8 | - 235 | + 245 | - 205 | - 65 |
| 2.0 | 17.6 | - 870 | + 5 | - 600 | - 488 |
| 3.0 | 26.4 | - 595 | - 285 | - 740 | - 540 |
| 4.0 | 35.2 | - 605 | - 775 | - 600 | - 660 |
| 5.0 | 44.0 | - 620 | - 745 | - 615 | - 660 |
| 6.0 | 52.8 | - 640 | - 655 | - 635 | - 643 |
| 8.0 | 70.4 | - 655 | - 670 | - 645 | - 657 |
| 10.0 | 88.0 | - 680 | - 685 | - 655 | - 673 |
| 15.0 | 132.0 | - 715 | - 720 | - 695 | - 707 |
| 20.0 | 176.0 | - 745 | - 745 | - 720 | - 737 |
| 25.0 | 220.0 | - 785 | - 785 | - 745 | - 773 |
| 30.0 | 264.0 | - 845 | -845 | - 785 | - 825 |
| 35.0 | 308.0 | - 885 | -1295 | - 835 | -1005 |
| 40.0 | 352.0 | - 805 | -1335 | -1320 | -1153 |
| 45.0 | 396.0 | -1335 | -1370 | -1350 | -1352 |
| 50.0 | 440.0 | -1355 | -1385 | -1380 | -1373 |

TABLE No.6

Data for Curve No.1 on Chart No. 2a, 2b, 2c,
and for Curve No.3 on Chart No.5 .
Sample preparation: cleaning cycle No.6 .
Apparent cathode area: 0.114 sq.in.

| Polariz. current, mA | Cathode current density mA/sq.in | Cathode Potential, mV | | | |
|---|---|---|---|---|---|
| | | 1 | 2 | 3 | Average |
| 0.0 | 0.0 | + 330 | + 255 | + 305 | + 296 |
| 0.5 | 4.4 | + 315 | + 175 | + 265 | + 252 |
| 1.0 | 8.8 | + 305 | + 115 | + 255 | + 225 |
| 2.0 | 17.6 | + 295 | + 5 | + 225 | + 175 |
| 3.0 | 26.4 | + 285 | + 205 | + 175 | + 222 |
| 4.0 | 35.2 | + 270 | - 780 | - 355 | - 288 |
| 5.0 | 44.0 | + 265 | - 745 | - 805 | - 428 |
| 6.0 | 52.8 | + 255 | - 760 | - 805 | - 437 |
| 8.0 | 70.4 | + 250 | - 760 | - 705 | - 405 |
| 10.0 | 88.0 | + 245 | - 785 | - 705 | - 415 |
| 15.0 | 132.0 | + 210 | - 770 | - 720 | - 427 |
| 20.0 | 176.0 | + 170 | - 705 | - 745 | - 427 |
| 25.0 | 220.0 | + 110 | - 735 | - 755 | - 460 |
| 30.0 | 264.0 | + 10 | - 770 | - 785 | - 515 |
| 35.0 | 308.0 | - 745 | - | - 820 | - 782 |
| 40.0 | 352.0 | - 670 | -1420 | -1405 | -1163 |
| 45.0 | 396.0 | - 745 | -1430 | -1430 | -1202 |
| 50.0 | 440.0 | -1405 | -1430 | -1435 | -1423 |

TABLE No.7

Data for Curve No.2 on Charts No. 2a, 2b, 2c, and for Curve No.3 on Chart No.6 .
Sample preparation: cleaning cycle No.7 .
Apparent cathode area: 0.114 sq.in.

| Polariz. current, mA | Cathode current density mA/sq.in | Cathode Potential, mV | | | |
|---|---|---|---|---|---|
| | | 1 | 2 | 3 | Average |
| 0.0 | 0.0 | + 330 | + 305 | + 245 | + 293 |
| 0.5 | 4.4 | + 295 | + 270 | + 155 | + 240 |
| 1.0 | 8.8 | + 270 | + 255 | + 80 | + 202 |
| 2.0 | 17.6 | + 260 | + 240 | - 85 | + 138 |
| 3.0 | 26.4 | + 250 | + 220 | - 755 | - 95 |
| 4.0 | 35.2 | + 235 | + 200 | - 755 | - 107 |
| 5.0 | 44.0 | + 220 | + 180 | - 755 | - 118 |
| 6.0 | 52.8 | + 205 | + 155 | - 770 | - 137 |
| 8.0 | 70.4 | + 165 | + 105 | - 785 | - 172 |
| 10.0 | 88.0 | + 130 | + 30 | - 785 | - 208 |
| 15.0 | 132.0 | - 45 | - 830 | - 820 | - 565 |
| 20.0 | 176.0 | - 730 | - 720 | - 895 | - 782 |
| 25.0 | 220.0 | - 720 | - 745 | - 670 | - 712 |
| 30.0 | 264.0 | - 740 | - 770 | - 685 | - 732 |
| 35.0 | 308.0 | - 760 | -1090 | - 720 | - 857 |
| 40.0 | 352.0 | - 845 | -1420 | -1435 | -1233 |
| 45.0 | 396.0 | -1420 | -1430 | -1435 | -1428 |
| 50.0 | 440.0 | -1445 | -1445 | -1445 | -1445 |

TABLE No.8

Data for Curve No.3 on Charts No. 2a, 2b, 2c, and for Curve No.3 on Chart No.7 .
Sample preparation: cleaning cycle No.8 .
Apparent cathode area: 0.114 sq.in.

| Polariz. current, mA | Cathode current density mA/sq.in | Cathode Potential, mV | | | |
|---|---|---|---|---|---|
| | | 1 | 2 | 3 | Average |
| 0.0 | 0.0 | + 305 | + 285 | + 345 | + 312 |
| 0.5 | 4.4 | + 275 | + 255 | + 240 | + 257 |
| 1.0 | 8.8 | + 245 | + 240 | + 115 | + 200 |
| 2.0 | 17.6 | + 225 | + 215 | - 85 | + 118 |
| 3.0 | 26.4 | + 215 | + 200 | - 545 | - 43 |
| 4.0 | 35.2 | + 195 | + 190 | - 605 | - 73 |
| 5.0 | 44.0 | + 180 | + 175 | - 620 | - 88 |
| 6.0 | 52.8 | + 155 | + 165 | - 630 | - 103 |
| 8.0 | 70.4 | + 95 | + 140 | - 645 | - 136 |
| 10.0 | 88.0 | + 40 | + 115 | - 655 | - 163 |
| 15.0 | 132.0 | - 595 | + 50 | - 685 | - 410 |
| 20.0 | 176.0 | - 670 | - 120 | - 720 | - 503 |
| 25.0 | 220.0 | - 770 | - 685 | - 745 | - 733 |
| 30.0 | 264.0 | - 835 | - 705 | - 795 | - 778 |
| 35.0 | 308.0 | -1370 | - 760 | -1345 | -1158 |
| 40.0 | 352.0 | -1445 | -1370 | -1405 | -1407 |
| 45.0 | 396.0 | -1470 | -1410 | -1430 | -1437 |
| 50.0 | 440.0 | -1485 | -1430 | -1440 | -1452 |

TABLE No.9

Data for Curve No.4 on Charts No. 2a, 2b, 2c,
and for Curve No.3 on Chart No.8 .
Sample preparation: cleaning cycle No.9 .
Apparent cathode area: o.114 sq.in.

| Polariz. current, mA | Cathode current density mA/sq.in | Cathode Potential, mV | | | |
|---|---|---|---|---|---|
| | | 1 | 2 | 3 | Average |
| o.o | 0.0 | + 245 | + 130 | + 230 | + 202 |
| 0.5 | 4.4 | + 80 | - 70 | - 85 | - 25 |
| 1.0 | 8.8 | - 45 | - 170 | - 195 | - 170 |
| 2.0 | 17.6 | - 245 | - 245 | - 850 | - 447 |
| 3.0 | 26.4 | - 750 | - 655 | - 655 | - 687 |
| 4.0 | 35.2 | - 620 | - 660 | - 635 | - 638 |
| 5.0 | 44.0 | - 610 | - 670 | - 635 | - 638 |
| 6.0 | 52.8 | - 610 | - 675 | - 620 | - 635 |
| 8.0 | 70.4 | - 625 | - 695 | - 630 | - 650 |
| 10.0 | 88.0 | - 630 | - 710 | - 630 | - 657 |
| 15.0 | 132.0 | - 645 | - 750 | - 650 | - 682 |
| 20.0 | 176.0 | - 660 | - 815 | - 650 | - 708 |
| 25.0 | 220.0 | - 685 | - 670 | - 685 | - 680 |
| 30.0 | 264.0 | - 710 | - 695 | - 610 | - 672 |
| 35.0 | 306.0 | - 795 | - 685 | - 735 | - 738 |
| 40.0 | 352.0 | -1405 | - 705 | - 795 | - 968 |
| 45.0 | 396.0 | -1425 | - 795 | -1395 | -1205 |
| 50.0 | 440.0 | -1435 | -1405 | -1410 | -1417 |

TABLE No.10

Data for Curve No.1 on Charts No. 3a, 3b, 3c,
and for Curve No.4 on Chart No.5 .
Sample preparation: cleaning cycle No.10 .
Apparent cathode area: 0.114 sq.in.

| Polariz. current, mA | Cathode current density mA/sq.in | Cathode Potential, mV | | | |
|---|---|---|---|---|---|
| | | 1 | 2 | 3 | Average |
| 0.0 | 0.0 | + 330 | + 230 | + 305 | + 288 |
| 0.5 | 4.4 | + 280 | + 145 | + 280 | + 234 |
| 1.0 | 8.8 | + 270 | + 100 | + 255 | + 208 |
| 2.0 | 17.6 | + 245 | + 5 | + 230 | + 160 |
| 3.0 | 26.4 | + 200 | - 145 | + 195 | + 83 |
| 4.0 | 35.2 | + 90 | - 745 | + 165 | - 163 |
| 5.0 | 44.0 | + 5 | - 695 | + 115 | - 192 |
| 6.0 | 52.8 | - 95 | - 695 | + 75 | - 238 |
| 8.0 | 70.4 | - 685 | - 720 | - 5 | - 470 |
| 10.0 | 88.0 | - 745 | - 735 | - 820 | - 766 |
| 15.0 | 132.0 | - 805 | - 770 | - 820 | - 797 |
| 20.0 | 176.0 | - 855 | - 820 | - 820 | - 830 |
| 25.0 | 220.0 | - 645 | - 830 | - 645 | - 706 |
| 30.0 | 264.0 | - 655 | - 845 | - 655 | - 717 |
| 35.0 | 308.0 | - 720 | -1195 | - 720 | - 878 |
| 40.0 | 352.0 | -1370 | -1295 | - 795 | -1153 |
| 45.0 | 396.0 | -1395 | -1355 | -1395 | -1382 |
| 50.0 | 440.0 | -1420 | -1395 | -1420 | -1413 |

TABLE No.11

Data for Curve No.2 on Charts No. 3a, 3b, 3c,
and for Curve No.4 on Chart No.6 .
Sample preparation: cleaning cycle No.11 .
Apparent cathode area: o.114 sq.in.

| Polariz. current, mA | Cathode current density mA/sq.in | Cathode Potential, mV | | | |
|---|---|---|---|---|---|
| | | 1 | 2 | 3 | Average |
| 0.0 | 0.0 | + 255 | + 295 | + 255 | + 268 |
| 0.5 | 4.4 | + 80 | + 265 | + 55 | + 133 |
| 1.0 | 8.8 | - 20 | + 255 | - 45 | + 63 |
| 2.0 | 17.6 | - 155 | + 205 | - 135 | - 28 |
| 3.0 | 26.4 | - 795 | + 55 | - 695 | - 478 |
| 4.0 | 35.2 | - 730 | - 70 | - 670 | - 490 |
| 5.0 | 44.0 | - 730 | - 770 | - 685 | - 728 |
| 6.0 | 52.8 | - 720 | - 745 | - 670 | - 712 |
| 8.0 | 70.4 | - 745 | - 745 | - 695 | - 728 |
| 10.0 | 88.0 | - 760 | - 745 | - 705 | - 737 |
| 15.0 | 132.0 | - 820 | - 795 | - 770 | - 795 |
| 20.0 | 176.0 | - 870 | - 870 | - 845 | - 862 |
| 25.0 | 220.0 | - 870 | - 805 | - 830 | - 835 |
| 30.0 | 264.0 | - 795 | - 670 | - 770 | - 745 |
| 35.0 | 308.0 | - 870 | - 685 | - 845 | - 800 |
| 40.0 | 352.0 | -1230 | - 745 | - 865 | - 947 |
| 45.0 | 396.0 | -1305 | -1345 | - 745 | -1132 |
| 50.0 | 440.0 | -1355 | -1420 | -1245 | -1340 |

TABLE No.12

Data for Curve No.3 on Charts No. 3a, 3b, 3c, and for Curve No.4 on Chart No.7 .
Sample preparation: cleaning cycle No.12 .
Apparent cathode area: 0.114 sq.in.

| Polariz. current, mA | Cathode current density mA/sq.in | Cathode Potential, mV | | | |
|---|---|---|---|---|---|
| | | 1 | 2 | 3 | Average |
| 0.0 | 0.0 | + 280 | + 330 | + 245 | + 285 |
| 0.5 | 4.4 | + 170 | + 255 | - 30 | + 132 |
| 1.0 | 8.8 | + 80 | + 155 | - 160 | + 25 |
| 2.0 | 17.6 | - 45 | - 45 | - 720 | - 270 |
| 3.0 | 26.4 | - 170 | - 170 | - 695 | - 344 |
| 4.0 | 35.2 | - 525 | - 555 | - 695 | - 592 |
| 5.0 | 44.0 | - 495 | - 575 | - 695 | - 588 |
| 6.0 | 52.8 | - 505 | - 555 | - 710 | - 590 |
| 8.0 | 70.4 | - 530 | - 575 | - 740 | - 615 |
| 10.0 | 88.0 | - 545 | - 575 | - 745 | - 622 |
| 15.0 | 132.0 | - 580 | - 605 | - 795 | - 660 |
| 20.0 | 176.0 | - 605 | - 635 | - 845 | - - 695 |
| 25.0 | 220.0 | - 630 | - 680 | - 920 | - 743 |
| 30.0 | 264.0 | - 685 | - 735 | - 905 | - 775 |
| 35.0 | 308.0 | - 745 | - 845 | - 785 | - 792 |
| 40.0 | 352.0 | -1270 | -1195 | - 805 | -1090 |
| 45.0 | 396.0 | -1330 | -1270 | - 850 | -1150 |
| 50.0 | 440.0 | -1375 | -1305 | - 945 | -1208 |

TABLE No.13

Data for Curve No.4 on Charts No. 3a, 3b, 3c,
and Curve No.4 on Chart No.8 .
Sample preparation: cleaning cycle No.13 .
Apparent cathode area: 0.114 sq.in.

| Polariz. current, mA | Cathode current density mA/sq.in | Cathode Potential, mV | | | |
|---|---|---|---|---|---|
| | | 1 | 2 | 3 | Average |
| 0.0 | 0.0 | + 230 | + 170 | + 280 | + 227 |
| 0.5 | 4.4 | - 45 | - 55 | + 235 | + 45 |
| 1.0 | 8.8 | - 135 | - 120 | + 180 | - 25 |
| 2.0 | 17.6 | - 245 | - 220 | + 85 | - 127 |
| 3.0 | 26.4 | - 745 | - 730 | - 35 | - 503 |
| 4.0 | 35.2 | - 695 | - 685 | - 245 | - 542 |
| 5.0 | 44.0 | - 695 | - 695 | - 645 | - 678 |
| 6.0 | 52.8 | - 695 | - 695 | - 645 | - 678 |
| 8.0 | 70.4 | - 725 | - 715 | - 655 | - 698 |
| 10.0 | 88.0 | - 740 | - 730 | - 660 | - 710 |
| 15.0 | 132.0 | - 820 | - 755 | - 700 | - 758 |
| 20.0 | 176.0 | - 880 | - 795 | - 745 | - 773 |
| 25.0 | 220.0 | - 695 | - 870 | - 795 | - 787 |
| 30.0 | 264.0 | - 695 | - 660 | - 895 | - 750 |
| 35.0 | 308.0 | - 735 | - 670 | - 845 | - 750 |
| 40.0 | 352.0 | - 795 | - 685 | - 770 | - 750 |
| 45.0 | 396.0 | -1345 | - 720 | -1220 | -1095 |
| 50.0 | 440.0 | -1380 | - 770 | -1270 | -1140 |

TABLE No.14

Data for Curve No.5 on Charts No. 2a, 2b, 2c, and for Curve No.2 on Chart No.9 .
Sample preparation: cleaning cycle No.14 .
Apparent cathode area: o.114 sq.in.

| Polariz. current, mA | Cathode current density mA/sq.in | Cathode Potential, mV | | | |
|---|---|---|---|---|---|
| | | 1 | 2 | 3 | Average |
| 0.0 | 0.0 | + 265 | + 165 | + 310 | + 245 |
| 0.5 | 4.4 | + 245 | + 30 | + 300 | + 192 |
| 1.0 | 8.8 | + 220 | - 55 | + 295 | + 152 |
| 2.0 | 17.6 | + 195 | - 245 | + 270 | + 73 |
| 3.0 | 26,4 | + 155 | - 750 | + 255 | - 113 |
| 4.0 | 35.2 | + 105 | - 695 | + 245 | - 115 |
| 5.0 | 44.0 | - 20 | - 705 | + 230 | - 165 |
| 6.0 | 52.8 | - 145 | - 695 | + 220 | - 207 |
| 8.0 | 70.4 | - 835 | - 720 | + 205 | - 450 |
| 10.0 | 88.0 | - 870 | - 720 | + 190 | - 466 |
| 15.0 | 132.0 | - 800 | - 755 | + 130 | - 475 |
| 20.0 | 176.0 | - 800 | - 785 | + 30 | - 518 |
| 25.0 | 220.0 | - 820 | - 805 | - 845 | - 823 |
| 30.0 | 264.0 | - 835 | - 835 | - 815 | - 828 |
| 35.0 | 308.0 | -1405 | -1410 | - 695 | -1170 |
| 40.0 | 352.0 | -1430 | -1425 | -,735 | -1195 |
| 45.0 | 396.0 | -1445 | -1430 | - 770 | -1215 |
| 50.0 | 440.0 | -1450 | -1435 | -1395 | -1430 |

TABLE No.15

Data for Curve No.6 on Charts No. 2a, 2b, 2c, and for Curve No.2 on Chart No.10 .
Sample preparation: cleaning cycle No.15 .
Apparent cathode area: 0.114 sq.in.

| Polariz. current, mA | Cathode current density mA/sq.in | Cathode Potential, mV | | | |
|---|---|---|---|---|---|
| | | 1 | 2 | 3 | Average |
| 0.0 | 0.0 | + 325 | + 180 | + 285 | + 233 |
| 0.5 | 4.4 | + 165 | - 105 | + 255 | + 105 |
| 1.0 | 8.8 | + 45 | - 165 | + 215 | + 32 |
| 2.0 | 17.6 | - 230 | - 245 | + 165 | - 103 |
| 3.0 | 26.4 | - 820 | - 795 | + 115 | - 515 |
| 4.0 | 35.2 | - 725 | - 690 | + 70 | - 448 |
| 5.0 | 44.0 | - 725 | - 690 | + 30 | - 462 |
| 6.0 | 52.8 | - 725 | - 675 | - 20 | - 473 |
| 8.0 | 70.4 | - 730 | - 675 | - 245 | - 550 |
| 10.0 | 88.0 | - 730 | - 685 | - 775 | - 730 |
| 15.0 | 132.0 | - 745 | - 705 | - 620 | - 690 |
| 20.0 | 176.0 | - 760 | - 720 | - 645 | - 708 |
| 25.0 | 220.0 | - 795 | - 740 | - 660 | - 732 |
| 30.0 | 264.0 | - 820 | - 760 | - 685 | - 755 |
| 35.0 | 308.0 | -1420 | - 795 | - 720 | - 978 |
| 40.0 | 352.0 | -1435 | - 1345 | - 770 | -1182 |
| 45.0 | 396.0 | -1450 | -1395 | -1395 | -1413 |
| 50.0 | 440.0 | -1460 | -1410 | -1420 | -1430 |

TABLE No.16

Data for Curve No.7 on Charts No. 2a, 2b, 2c, and for Curve No.2 on Chart No.11 .
Sample preparation: cleaning cycle No.16 .
Apparent cathode area: 0.114 sq.in.

| Polariz. current, mA | Cathode current density mA/sq.in | Cathode Potential, mV | | | |
|---|---|---|---|---|---|
| | | 1 | 2 | 3 | Average |
| 0.0 | 0.0 | + 115 | + 65 | + 55 | + 78 |
| 0.5 | 4.4 | - 105 | -155 | - 195 | - 152 |
| 1.0 | 8.8 | - 180 | - 245 | - 245 | - 223 |
| 2.0 | 17.6 | - 745 | - 770 | - 645 | - 720 |
| 3.0 | 26.4 | - 785 | - 705 | - 645 | - 712 |
| 4.0 | 35.2 | - 745 | - 705 | - 650 | - 700 |
| 5.0 | 44.0 | - 745 | - 720 | - 650 | - 705 |
| 6.0 | 52.8 | - 750 | - 720 | - 660 | - 710 |
| 8.0 | 70.4 | - 765 | - 740 | - 680 | - 728 |
| 10.0 | 88.0 | - 775 | - 745 | - 685 | - 735 |
| 15.0 | 132.0 | - 810 | - 770 | - 720 | - 767 |
| 20.0 | 176.0 | - 830 | - 795 | - 750 | - 790 |
| 25.0 | 220.0 | - 845 | - 820 | - 785 | - 817 |
| 30.0 | 264.0 | - 845 | - 845 | - 805 | - 832 |
| 35.0 | 308.0 | - 870 | -1405 | - 845 | -1040 |
| 40.0 | 352.0 | -1370 | -1425 | -1385 | -1393 |
| 45.0 | 396.0 | -1395 | -1435 | -1410 | -1413 |
| 50.0 | 440.0 | -1410 | -1445 | -1420 | -1425 |

TABLE No.17

Data for Curve No.8 on Charts No. 2a, 2b, 2c, and for Curve No.2 on Chart No.12 .
Sample preparation: cleaning cycle No.17 .
Apparent cathode area: 0.114 sq.in.

| Polariz. current, mA | Cathode current density mA/sq.in | Cathode Potential, mV | | | |
|---|---|---|---|---|---|
| | | 1 | 2 | 3 | Average |
| 0.0 | 0.0 | + 245 | + 180 | + 240 | + 222 |
| 0.5 | 4.4 | - 95 | - 5 | + 145 | + 15 |
| 1.0 | 8.8 | - 205 | - 105 | + 65 | - 82 |
| 2.0 | 17.6 | - 815 | - 245 | - 35 | - 365 |
| 3.0 | 26.4 | - 745 | - 805 | - 155 | - 568 |
| 4.0 | 35.2 | - 735 | - 770 | - 775 | - 760 |
| 5.0 | 44.0 | - 720 | - 775 | - 655 | - 717 |
| 6.0 | 52.8 | - 710 | - 765 | - 655 | - 710 |
| 8.0 | 70.4 | - 725 | - 785 | - 655 | - 722 |
| 10.0 | 88.0 | - 725 | - 795 | - 655 | - 725 |
| 15.0 | 132.0 | - 735 | - 835 | - 680 | - 750 |
| 20.0 | 176.0 | - 755 | - 720 | - 695 | - 723 |
| 25.0 | 220.0 | - 770 | - 705 | - 705 | - 727 |
| 30.0 | 264.0 | - 795 | - 710 | - 720 | - 742 |
| 35.0 | 308.0 | - 820 | - 730 | - 745 | - 765 |
| 40.0 | 352.0 | -1370 | - 760 | - 795 | - 975 |
| 45.0 | 396.0 | -1395 | - 845 | - 870 | -1037 |
| 50.0 | 440.0 | -1410 | -1395 | -1405 | -1403 |

TABLE No.18

Data for Curve No.5 on Charts No. 3a, 3b, 3c,
and for Curve No.3 on Chart No.9 .
Sample preparation: cleaning cycle No.18 .
Apparent cathode area: 0.114 sq.in.

| Polariz. current, mA | Cathode current density mA/sq.in | Cathode Potential, mV | | | |
|---|---|---|---|---|---|
| | | 1 | 2 | 3 | Average |
| 0.0 | 0.0 | + 345 | + 315 | + 310 | + 323 |
| 0.5 | 4.4 | + 295 | + 270 | + 265 | + 277 |
| 1.0 | 8.8 | + 265 | + 245 | + 245 | + 252 |
| 2.0 | 17.6 | + 215 | + 205 | + 195 | + 205 |
| 3.0 | 26.4 | + 145 | + 155 | + 105 | + 135 |
| 4.0 | 35.2 | + 70 | + 65 | - 55 | + 27 |
| 5.0 | 44.0 | - 5 | - 45 | - 180 | - 77 |
| 6.0 | 52.8 | - 245 | - 220 | - 270 | - 245 |
| 8.0 | 70.4 | - 705 | - 710 | - 750 | - 722 |
| 10.0 | 88.0 | - 705 | - 705 | - 705 | - 705 |
| 15.0 | 132.0 | - 720 | - 725 | - 725 | - 723 |
| 20.0 | 176.0 | - 740 | - 740 | - 745 | - 742 |
| 25.0 | 220.0 | - 755 | - 750 | - 770 | - 758 |
| 30.0 | 264.0 | - 780 | - 780 | - 805 | - 788 |
| 35.0 | 308.0 | - 785 | - 820 | - 845 | - 817 |
| 40.0 | 352.0 | - 845 | - 720 | -1270 | - 945 |
| 45.0 | 396.0 | -1195 | - 795 | -1305 | -1097 |
| 50.0 | 440.0 | -1245 | -1385 | -1345 | -1325 |

TABLE No.19

Data for Curve No.6 on Charts No. 3a, 3b, 3c,
and Curve No.3 on Chart No.10 .
Sample preparation: cleaning cycle No.19 .
Apparent cathode area: 0.114 sq.in.

| Polariz. Current, mA | Cathode current density mA/sq.in | Cathode Potential, mV | | | |
|---|---|---|---|---|---|
| | | 1 | 2 | 3 | Average |
| 0.0 | 0.0 | + 155 | + 245 | + 230 | + 210 |
| 0.5 | 4.4 | - 45 | + 35 | + 95 | + 28 |
| 1.0 | 8.8 | - 130 | - 45 | + 5 | - 57 |
| 2.0 | 17.6 | - 245 | - 220 | - 135 | - 200 |
| 3.0 | 26.4 | - 585 | - 635 | - 245 | - 488 |
| 4.0 | 35.2 | - 585 | - 645 | - 685 | - 638 |
| 5.0 | 44.0 | - 595 | - 645 | - 665 | - 635 |
| 6.0 | 52.8 | - 600 | - 645 | - 660 | - 635 |
| 8.0 | 70.4 | - 610 | - 655 | - 660 | - 642 |
| 10.0 | 88.0 | - 630 | - 655 | - 660 | - 648 |
| 15.0 | 132.0 | - 645 | - 670 | - 675 | - 663 |
| 20.0 | 176.0 | - 660 | - 695 | - 695 | - 683 |
| 25.0 | 220.0 | - 695 | - 715 | - 710 | - 707 |
| 30.0 | 264.0 | - 720 | - 745 | - 735 | - 733 |
| 35.0 | 308.0 | - 770 | - 795 | - 760 | - 775 |
| 40.0 | 352.0 | - 845 | -1250 | - 870 | - 988 |
| 45.0 | 396.0 | -1270 | -1345 | - 745 | -1120 |
| 50.0 | 440.0 | -1385 | -1385 | -1345 | -1371 |

TABLE No.20

Data for Curve No.7 on Charts No. 3a, 3b, 3c,
and for Curve No.3 on Chart No.11 .
Sample preparation: cleaning cycle No.20 .
Apparent cathode area: 0.114 sq.in.

| Polariz. current, mA | Cathode current density mA/sq.in | Cathode Potential, mV | | | |
|---|---|---|---|---|---|
| | | 1 | 2 | 3 | Average |
| 0.0 | 0.0 | + 130 | + 155 | + 235 | + 173 |
| 0.5 | 4.4 | - 120 | - 95 | - 35 | - 83 |
| 1.0 | 8.8 | - 170 | - 155 | - 145 | - 156 |
| 2.0 | 17.6 | - 245 | - 245 | - 245 | - 245 |
| 3.0 | 26.4 | - 755 | - 710 | - 765 | - 743 |
| 4.0 | 35.2 | - 695 | - 655 | - 705 | - 685 |
| 5.0 | 44.0 | - 695 | - 655 | - 710 | - 687 |
| 6.0 | 52.8 | - 695 | - 660 | - 705 | - 687 |
| 8.0 | 70.4 | - 705 | - 675 | - 720 | - 700 |
| 10.0 | 88.0 | - 710 | - 685 | - 720 | - 705 |
| 15.0 | 132.0 | - 730 | - 705 | - 745 | - 727 |
| 20.0 | 176.0 | - 750 | - 725 | - 760 | - 745 |
| 25.0 | 220.0 | - 775 | - 745 | - 785 | - 768 |
| 30.0 | 264.0 | - 820 | - 775 | - 820 | - 805 |
| 35.0 | 308.0 | -870 | - 820 | - 870 | - 853 |
| 40.0 | 352.0 | - 970 | - 870 | - 895 | - 912 |
| 45.0 | 396.0 | - 870 | -1355 | - 745 | - 990 |
| 50.0 | 440.0 | -1345 | -1385 | - 820 | -1183 |

TABLE No.21

Data for Curve No.8 on Charts No. 3a, 3b, 3c, and for Curve No.3 on Chart No.12 .
Sample preparation: cleaning cycle No.21 .
Apparent cathode area: 0.114 sq.in.

| Polariz. current, mA | Cathode current density mA/sq.in | Cathode Potential, mV | | | |
|---|---|---|---|---|---|
| | | 1 | 2 | 3 | Average |
| 0.0 | 0.0 | + 130 | + 170 | + 230 | + 177 |
| 0.5 | 4.4 | - 95 | - 95 | - 45 | - 78 |
| 1.0 | 8.8 | - 165 | - 195 | - 145 | - 168 |
| 2.0 | 17.6 | - 245 | - 765 | - 245 | - 418 |
| 3.0 | 26.4 | - 730 | - 710 | - 770 | - 737 |
| 4.0 | 35.2 | - 705 | - 715 | - 695 | - 705 |
| 5.0 | 44.0 | - 705 | - 725 | - 695 | - 708 |
| 6.0 | 52.8 | - 705 | - 725 | - 695 | - 708 |
| 8.0 | 70.4 | - 720 | - 745 | - 705 | - 723 |
| 10.0 | 88.0 | - 725 | - 745 | - 705 | - 725 |
| 15.0 | 132.0 | - 745 | - 775 | - 740 | - 753 |
| 20.0 | 176.0 | - 775 | - 810 | - 750 | - 778 |
| 25.0 | 220.0 | - 795 | - 845 | - 780 | - 807 |
| 30.0 | 264.0 | - 835 | - 870 | - 810 | - 838 |
| 35.0 | 308.0 | - 870 | - 945 | - 870 | - 895 |
| 40.0 | 352.0 | - 945 | - 845 | -1245 | -1012 |
| 45.0 | 396.0 | -1020 | - 845 | -1355 | -1073 |
| 50.0 | 440.0 | -1145 | -1095 | -1385 | -1208 |

# TABLE No.22

Data for Curve No.1 on Charts No. 4a, 4b, 4c,
Sample preparation: cleaning cycle No.7 .
Apparent cathode area: o.114 sq.in.

| Polariz. current, mA | Cathode current density mA/sq.in | Cathode Potential, mV | | | | |
|---|---|---|---|---|---|---|
| | | 1 | 2 | 3 | 4 | Average |
| 0.0 | 0.0 | + 405- | + 415 | + 400 | + 475 | + 424 |
| 0.5 | 4.4 | + 340 | + 365 | + 395 | + 355 | + 364 |
| 1.0 | 8.8 | + 305 | + 345 | + 380 | + 305 | + 304 |
| 2.0 | 17.6 | + 95 | + 305 | + 360 | + 30 | + 198 |
| 3.0 | 26.4 | - 245 | + 165 | + 350 | - 130 | + 35 |
| 4.0 | 35.2 | - 705 | + 105 | + 340 | - 645 | - 226 |
| 5.0 | 44.0 | - 695 | + 5 | + 330 | - 655 | - 288 |
| 6.0 | 52.8 | - 690 | - 840 | + 315 | - 655 | - 468 |
| 8.0 | 70.4 | - 695 | - 620 | + 295 | - 675 | - 399 |
| 10.0 | 88.0 | - 695 | - 595 | + 280 | - 685 | - 424 |
| 15.0 | 132.0 | - 715 | - 635 | + 155 | - 730 | - 481 |
| 20.0 | 176.0 | -1310 | - 655 | - 745 | - 845 | - 889 |
| 25.0 | 220.0 | -1355 | - 700 | -1245 | -1305 | -1151 |
| 30.0 | 264.0 | -1385 | -1375 | -1370 | -1370 | -1375 |
| 35.0 | 308.0 | -1395 | -1395 | -1405 | -1410 | -1401 |
| 40.0 | 352.0- | -1400 | -1400 | -1420 | -1425 | -1411 |
| 45.0 | 396.0 | -1410 | -1410 | -1435 | -1435 | -1422 |
| 50.0 | 440.0 | -1420 | -1420 | -1445 | -1445 | -1432 |

## TABLE No.23

Data for Curve No.2 on Charts No. 4a, 4b, 4c
Sample preparation: cleaning cycle No.11 .
Apparent cathode area: 0.114 sq.in.

| Polariz. Current, mA | Cathode current density mA/sq.in | Cathode Potential, mV | | | | |
|---|---|---|---|---|---|---|
| | | 1 | 2 | 3 | 4 | Average |
| 0.0 | 0.0 | + 405 | + 400 | + 415 | + 365 | + 396 |
| 0.5 | 4.4 | + 390 | + 245 | + 355 | + 335 | + 331 |
| 1.0 | 8.8 | + 365 | + 65 | + 330 | + 320 | + 270 |
| 2.0 | 17.6 | + 345 | - 470 | + 295 | + 290 | + 115 |
| 3.0 | 26.4 | + 330 | - 695 | + 265 | + 250 | + 37 |
| 4.0 | 35.2 | + 315 | - 695 | + 130 | + 40 | - 52 |
| 5.0 | 44.0 | + 305 | - 695 | - 155 | - 295 | - 210 |
| 6.0 | 52.8 | + 295 | - 705 | - 885 | - 785 | - 520 |
| 8.0 | 70.0 | + 265 | - 710 | - 655 | - 760 | - 465 |
| 10.0 | 88.0 | + 245 | - 735 | - 650 | - 650 | - 770 |
| 15.0 | 132.0 | + 145 | - 660 | - 680 | - 680 | - 468 |
| 20.0 | 176.0 | - 785 | - 645 | - 720 | - 705 | - 714 |
| 25.0 | 220.0 | - 655 | - 675 | - 720 | - 820 | - 717 |
| 30.0 | 264.0 | - 705 | -1280 | - 895 | -1220 | -1187 |
| 35.0 | 308.0 | -1275 | -1360 | -1290 | -1240 | -1291 |
| 40.0 | 352.0 | -1370 | -1385 | -1380 | -1370 | -1376 |
| 45.0 | 396.0 | -1395 | -1395 | -1395 | -1410 | -1399 |
| 50.0 | 440.0 | -1405 | -1395 | -1405 | -1430 | -1409 |

TABLE No.24

Data for Curve No.3 on Charts No. 4a, 4b, 4c .
Sample preparation: cleaning Cycle No.1 .
Apparent cathode area: 0.114 sq.in.

| Polariz. current, mA | Cathode current density mA/sq.in | Cathode Potential, mV | | | | |
|---|---|---|---|---|---|---|
| | | 1 | 2 | 3 | 4 | Average |
| 0.0 | 0.0 | + 445 | + 480 | + 365 | + 385 | + 419 |
| 0.5 | 4.4 | + 310 | + 370 | + 355 | + 365 | + 350 |
| 1.0 | 8.8 | + 245 | + 345 | + 340 | + 375 | + 327 |
| 2.0 | 17.6 | + 55 | + 250 | + 315 | + 245 | + 217 |
| 3.0 | 26.4 | - 805 | + 105 | + 285 | + 5 | - 102 |
| 4.0 | 35.2 | - 775 | - 855 | + 260 | - 395 | - 444 |
| 5.0 | 44.0 | - 785 | - 700 | + 220 | - 715 | - 495 |
| 6.0 | 52.8 | - 800 | - 710 | + 170 | - 705 | - 511 |
| 8.0 | 70.4 | - 795 | - 735 | + 75 | - 745 | - 550 |
| 10.0 | 88.0 | - 830 | - 750 | - 495 | - 745 | - 705 |
| 15.0 | 132.0 | -1370 | - 770 | - 700 | - 800 | - 910 |
| 20.0 | 176.0 | -1400 | -1260 | - 750 | -1275 | -1171 |
| 25.0 | 220.0 | -1410 | -1375 | - 785 | -1360 | -1233 |
| 30.0 | 264.0 | -1420 | -1395 | -770 | -1395 | -1245 |
| 35.0 | 308.0 | -1435 | -1415 | -1295 | -1410 | -1389 |
| 40.0 | 352.0 | -1445 | -1430 | -1340 | -1430 | -1411 |
| 45.0 | 396.0 | -1450 | -1445 | -1355 | -1445 | -1424 |
| 50.0 | 440.0 | -1455 | -1450 | -1410 | -1450 | -1441 |

TABLE No.25

Data for Curve No.4 on Charts No. 4a, 4b, 4c .
Sample preparation: cleaning cycle No.3 .
Apparent cathode area: 0.114 sq.in.

| Polariz. current, mA | Cathode current density mA/sq.in | Cathode Potential, mV | | | | |
|---|---|---|---|---|---|---|
| | | 1 | 2 | 3 | 4 | Average |
| 0.0 | 0.0 | + 555 | + 455 | + 930 | + 415 | + 589 |
| 0.5 | 4.4 | + 395 | + 330 | + 205 | + 365 | + 324 |
| 1.0 | 8.8 | + 385 | + 115 | - 5 | + 345 | + 210 |
| 2.0 | 17.6 | + 360 | - 745 | - 345 | + 315 | - 104 |
| 3.0 | 26.4 | + 330 | - 685 | - 760 | + 295 | - 205 |
| 4.0 | 35.2 | + 295 | - 695 | - 750 | + 270 | - 220 |
| 5.0 | 44.0 | + 225 | - 705 | - 755 | + 245 | - 247 |
| 6.0 | 52.0 | + 170 | - 700 | - 755 | + 205 | - 270 |
| 8.0 | 70.4 | + 95 | - 720 | - 770 | + 105 | - 323 |
| 10.0 | 88.0 | - 520 | - 725 | - 770 | - 45 | - 515 |
| 15.0 | 132.0 | - 720 | - 705 | - 795 | - 705 | - 731 |
| 20.0 | 176.0 | - 945 | -1300 | -1285 | -1300 | -1207 |
| 25.0 | 220.0 | -1330 | -1370 | -1360 | -1380 | -1360 |
| 30.0 | 264.0 | -1385 | -1395 | -1390 | -1400 | -1392 |
| 35.0 | 308.0 | -1405 | -1415 | -1395 | -1420 | -1409 |
| 40.0 | 352.0 | -1425 | -1435 | -1410 | -1440 | -1427 |
| 45.0 | 396.0 | -1430 | -1445 | -1430 | -1445 | -1437 |
| 50.0 | 440.0 | -1440 | -1450 | -1445 | -1455 | -1447 |

TABLE No.26

Data for Curve No.5 on Charts No. 4a, 4b, 4c .
Sample preparation: cleaning cycle No.4 .
Apparent cathode area: 0.114 sq.in.

| Polariz. current, mA | Cathode current density mA/sq.in | Cathode Potential, mV | | | | |
|---|---|---|---|---|---|---|
| | | 1 | 2 | 3 | 4 | Average |
| 0.0 | 0.0 | + 415 | + 395 | + 345 | + 375 | + 382 |
| 0.5 | 4.4 | + 345 | + 330 | + 335 | + 355 | + 341 |
| 1.0 | 8.8 | + 305 | + 275 | + 315 | + 345 | + 310 |
| 2.0 | 17.6 | + 205 | + 205 | + 285 | + 320 | + 254 |
| 3.0 | 26.4 | + 105 | + 135 | + 265 | + 305 | + 202 |
| 4.0 | 35.2 | - 35 | + 55 | + 225 | + 285 | + 132 |
| 5.0 | 44.0 | - 755 | - 130 | + 185 | + 265 | - 109 |
| 6.0 | 52.8 | - 755 | - 685 | + 155 | + 245 | - 260 |
| 8.0 | 70.4 | - 740 | - 615 | + 90 | + 190 | - 269 |
| 10.0 | 88.0 | - 735 | - 595 | - 5 | + 125 | - 302 |
| 15.0 | 132.0 | - 785 | - 610 | - 680 | - 645 | - 680 |
| 20.0 | 176.0 | -1330- | - 635 | - 710 | - 785 | - 865 |
| 25.0 | 220.0 | -1375 | -1260 | - 750 | -1390 | -1194 |
| 30.0 | 264.0 | -1400 | -1350 | - 790 | -1420 | -1240 |
| 35.0 | 308.0 | -1410 | -1385 | -1285 | -1440 | -1380 |
| 40.0 | 352.0 | -1410 | -1400 | -1315 | -1455 | -1395 |
| 45.0 | 396.0 | -1425 | -1420 | -1345 | -1470 | -1415 |
| 50.0 | 440.0 | -1430 | -1435 | -1360 | -1480 | -1426 |

TABLE No.27

Data for Curve No.6 on Charts No. 4a, 4b, 4c .
Sample preparation: cleaning cycle No.5 .
Apparent cathode area: 0.114 sq.in.

| Polariz. current, mA | Cathode current density mA/sq.in | Cathode Potential, mV 1 | 2 | 3 | 4 | Average |
|---|---|---|---|---|---|---|
| 0.0 | 0.0 | + 420 | + 455 | +1005 | + 980 | + 715 |
| 0.5 | 4.4 | +380 | + 305 | + 5 | + 30 | + 180 |
| 1.0 | 8.8 | + 360 | - 320 | - 295 | - 855 | - 278 |
| 2.0 | 17.6 | + 330 | - 590 | - 895 | - 570 | - 431 |
| 3.0 | 26.4 | + 305 | - 580 | - 560 | - 580 | - 354 |
| 4.0 | 35.2 | + 280 | - 655 | - 570 | - 605 | - 387 |
| 5.0 | 44.0 | + 250 | - 670 | - 585 | - 625 | - 407 |
| 6.0 | 52.8 | + 220 | - 685 | - 600 | - 635 | - 425 |
| 8.0 | 70.4 | + 155 | - 695 | - 635 | - 660 | - 459 |
| 10.0 | 88.0 | + 100 | - 695 | - 650 | - 675 | - 480 |
| 15.0 | 132.0 | - 595 | - 750 | - 705 | - 705 | - 689 |
| 20.0 | 176.0 | -1185 | -1320 | - 820 | - 845 | -1042 |
| 25.0 | 220.0 | -1335 | -1380 | - 895 | -1355 | -1241 |
| 30.0 | 264.0 | -1360 | -1385 | -1390 | -1385 | - 1380 |
| 35.0 | 308.0 | -1390 | -1400 | -1410 | -1405 | -1401 |
| 40.0 | 352.0 | -1400 | -1405 | -1435 | -1430 | -1417 |
| 45.0 | 396.0 | -1405 | -1420 | -1455 | -1445 | -1431 |
| 50.0 | 440.0 | -1410 | -1430 | -1465 | -1445 | -1438 |

TABLE No.28

Validity test data.

| Sample preparation | Cleaning cycle number 2 | 6 | 13 |
|---|---|---|---|
| Sample No.1 | | | |
| Weight plated, g | 0.5887 | 0.5855 | 0.5873 |
| Weight stripped, g | 0.5877 | 0.5841 | 0.5868 |
| Weight of Cr , g | 0.0010 | 0.0014 | 0.0005 |
| Sample No.2 | | | |
| Weight plated, g | 0.5870 | 0.5880 | 0.5901 |
| Weight stripped, g | 0.5855 | 0.5872 | 0.5897 |
| Weight of Cr , g | 0.0015 | 0.0008 | 0.0004 |
| Sample No.3 | | | |
| Weight plated, g | 0.5899 | 0.5897 | 0.5904 |
| Weight stripped, g | 0.5888 | 0.5887 | 0.5894 |
| Weight of Cr , g | 0.0011 | 0.0010 | 0.0010 |
| Average weight of Cr on three samples, g | 0.0012 | 0.0011 | 0.0006 |
| Average thickness of Cr on three samples, mm | 0.0227 | 0.0203 | 0.0119 |

## CLEANING SCHEDULES

| Cycle No. | Description of Cycle |
|---|---|
| 1 | Sample polished with dry sandpaper; solvent degreased. |
| 2 | Sample polished with dry sandpaper; solvent degreased; wet-scoured; rinsed. |
| 3 | Sample polished with dry sandpaper; solvent degreased; soaked in alkaline cleaner; rinsed. |
| 4 | Sample polished with dry sandpaper; solvent degreased; electrocleaned cathodically; rinsed. |
| 5 | Sample polished with dry sandpaper; solvent degreased; electrocleaned anodically; rinsed. |
| 6 | Sample polished with dry sandpaper; solvent degreased; pickled in HCl; rinsed. |
| 7 | Sample polished with dry sandpaper; solvent degreased; alkali soak-cleaned; rinsed; pickled in HCl; rinsed. |
| 8 | Sample polished with dry sandpaper; solvent degreased; electrocleaned cathodically; rinsed; pickled in HCl ; rinsed. |
| 9 | Sample polished with dry sandpaper; solvent degreased; electrocleaned anodically; rinsed; pickled in HCl ; rinsed. |
| 10 | Sample polished with dry sandpaper; solvent degreased; pickled in $H_2SO_4$ ; rinsed. |
| 11 | Sample polished with dry sandpaper; solvent degreased; alkali soak-cleaned; rinsed; pickled in $H_2SO_4$; rinsed. |
| 12 | Sample polished with dry sandpaper; solvent degreased; electrocleaned cathodically; rinsed; pickled in $H_2SO_4$; rinsed. |
| 13 | Sample polished with dry sandpaper; solvent degreased; electrocleaned anodically; rinsed; pickled in $H_2SO_4$; rinsed. |
| 14 | Sample polished with dry sandpaper; solvent degreased; electrocleaned cathodically; rinsed; pickled in HCl ; rinsed; electrocleaned cathodically; rinsed; pickled in HCl; rinsed. |

| Cycle No. | Description of Cycle |
|---|---|
| 15 | Sample polished with dry sandpaper; solvent degreased; electrocleaned anodically; rinsed; pickled in HCl ; rinsed; electrocleaned cathodically; rinsed; pickled in HCl; rinsed. |
| 16. | Sample polished with dry sandpaper; solvent degreased; electrocleaned cathodically; rinsed; pickled in HCl ; rinsed; electrocleaned anodically; rinsed; pickled in HCl; rinsed. |
| 17 | Sample polished with dry sandpaper; solvent degreased; electrocleaned anodically; rinsed; pickled in HCl ; rinsed; electrocleaned anodically; rinsed; pickled in HCl; rinsed. |
| 18 | Sample polished with dry sandpaper; solvent degreased; electrocleaned cathodically; rinsed; pickled in HCl ; rinsed; electrocleaned cathodically; rinsed; pickled in $H_2SO_4$ ; rinsed. |
| 19 | Sample polished with dry sandpaper; solvent degreased; electrocleaned anodically; rinsed; pickled in HCl ; rinsed; electrocleaned cathodically; rinsed; pickled in $H_2SO_4$ ; rinsed. |
| 20 | Sample polished with dry sandpaper; solvent degreased; electrocleaned cathodically; rinsed; pickled in HCl ; rinsed; electrocleaned anodically; rinsed; pickled in $H_2SO_4$ ; rinsed. |
| 21 | Sample polished with dry sandpaper; solvent degreased; electrocleaned anodically; rinsed; pickled in HCl ; rinsed; electrocleaned anodically; rinsed; pickled in $H_2SO_4$ ; rinsed. |

## DETAILS OF INDIVIDUAL STEPS USED IN PREPARATION OF SAMPLES

1. Sandpaper polishing.

Samples were rubbed(lengthwise and circumferentially) with Carborundum Co.'s Extra Fine Indian Head Flint Paper, until a uniform luster was obtained.

2. Solvent degreasing.

Samples were agitated in 1,1,1,-trichloroethane for 30 seconds; excess solvent was shaken off and samples were air-dried before next step.

3. Alkaline soak cleaning.

Samples were immersed for two minutes into the following bath:

| | |
|---|---|
| Caustic Soda | 16 g/l |
| Sodium Metasilicate | 8 g/l |
| Sodium Carbonate | 8 g/l |
| Trisodium Phosphate | 8 g/l |

( Operating temperature: 70 to 85°C )

4. Electrocleaning.

Both the cathodic and the anodic electrocleaning were performed by treating samples for two minutes in a bath of the following composition;

| | |
|---|---|
| Caustic Soda | 75 g/l |
| Trisodium Phosphate | 10 g/l |
| Sodium Carbonate | 14 g/l |

Counter-electrodes- low-carbon steel.
Temperature: 70 to 85°C
Current density (at the sample): 100 asf

5. Hydrochloric acid pickling.

Samples were kept for three minutes in a bath made by mixing one volume of commercial muriatic acid with two volumes of water. Bath was used at room temperature, without agitation.

6. Sulfuric acid pickling.

Samples were kept for three minutes in a bath made by mixing one volume of commercial concentrated $H_2SO_4$ with four volumes of water. Bath was used at room temperature, without agitation.

7. Wet scouring.

This operation was performed by rubbing samples for 30 to 60 seconds with a wet cloth pad, dipped into a household scouring powder ("Ajax"). Excess powder was flushed off with tap water before the sample was subjected to the rinsing operation proper.

8. Rinsing.

A three-stage rinse was employed. The first two stages comprised tap water, but distilled water was used in the final rinse. In all cases, samples were rinsed for 30 seconds, with a continuous agitation.

## APPARATUS

1. Modified*) EICO Model 214 vacuum-tube voltmeter, 22 megohm input resistance.
2. Lafayette Model 30-A volt-ohmmeter, 20,000 ohms per volt.
3. Saturated calomel electrode, Beckman.
4. Stirrer assembly, consisting of a three-speed motor, a flexible shaft, and a loop impeller.
5. Dry cells, 6 volt Eveready Heavy Duty No.509.
6. Storage battery, 6 volt, 70 ampere-hours Allstate No.57 .
7. Electrical connectors: plugs, wires, terminal posts, alligator clips, switches.
8. Potentiometer - 1,000 ohm, 2 watt; resistor - 150 ohm, 2 watt; rheostat - 600 ohm, 2 watt .
9. Electric hot plate.
10. Polyvinyl chloride tubing and stop-off tape .
11. Chemicals: NaOH, $Na_2CO_3$, $NaSiO_3$, $Na_3PO_4$, HCl, $H_2SO_4$ , 1,1,1 - trichloroethane, distilled water, CR-110 (a self-regulating chromium plating compound, made by the United Chromium Co.).
12. High-carbon steel samples.
13. Low-carbon steel strip for anodes.

*) The modification comprised:

1. Expanding the lowest available "DC Volts" scale by a factor of two.
2. Building into the meter a solenoid-actuated chopper bar for arresting pointer at regular intervals to facilitate the reading of a fluctuating voltage.

# DISCUSSION

Selection of the procedure and of electrolytes to be used in conducting the experiment was made on the basis of preliminary trial tests or on the basis of certain considerations as explained below.

## A. Haring's Method

The possibility of using Haring's method of potential measurement (43), which requires the use of a reference electrode made from the same material as the cathode, was investigated. Only a few runs were made and the method was abandoned for lack of reproducibility.

## B. Speed of Agitation

Various stirrer speeds, from zero speed (no agitation) to the maximum speed obtainable with the available multi-speed agitator motor, were tried. It was found that when no agitation or slow-speed agitation was employed, the potential stabilization occured rather slowly and that reproducibility of readings was poor. On the other hand, when the stirrer was run at a maximum speed, too much splashing occured and the electrolyte was visibly aerated. Splashing covered the electrode carrier arm with the electrolyte and this created a current leackage path and a possibility of erroneous readings. The medium stirrer speed (approximately 100 rpm) was, therefore, selected as the standard agi -

tation. This was fast enough to provide a noticeable flow of electrolyte past the cathode, yet did not cause undue splashing or aeration.

## C. Calomel Electrode

Several methods of connecting the calomel electrode to the electrolysis cell were tried. In each case, electrode was surrounded with a KCl-filled glass jacket (see Fig. 5) which was drawn to a capillary tip. When the top of jacket was tightly closed with a greased rubber stopper, the level of KCl solution within remained constant. It was noted however, that as soon as the tip of the jacket touched surface of the $CrO_3$ electrolyte, the latter would diffuse upward into the KCl at an amazingly fast rate. A cotton plug, inserted into the capillary, failed to completely stop this diffusion. Finally, the diffusion of the $CrO_3$ solution was counterbalanced by a controlled bleeding of the KCl solution from the capillary tip of the jacket. However, potential of the cathode measured with this arrangement proved to vary in an unpredictable fashion, and at higher densities of the polarizing current a chlorine odor began to emanate from the electrolysis cell. Samples, when removed from the solution, were also found to have this odor. Evidently, the effluent KCl was being decomposed and was causing fluctuations of the potential. Since the bleeding method of stopping diffusion of the $CrO_3$ could not be used, several more attempts to use cotton and asbestos plugs were made. These were finally replaced by a plug made from gelatin containing about 5% of KCl. The gelatin plug proved to be most satisfactory and completely stopped the diffusion of $CrO_3$ into the KCl solution.

## D. Concentration of the Electrolyte

The ease of handling of the experimental setup was tested with NaCl and KCl electrolytes of various concentrations; the cathode potential readings were not recorded. Since the decision was made beforehand that the experiment was to be run with CR-110,- a commercial chromium plating formulation, this was tested in four concentration: full-strength and diluted to be approximately 1.0 M, 0.1 M, and 0.01 M in $CrO_3$. While all of these performed satisfactorily, the undiluted solution (about 250 g/l of $CrO_3$) was too opaque to permit a visual examination of the surface of submerged electrodes. The 0.01 M solution was, of course, the most transparent,but its limitation was the possibility of concentration changes occuring during a lengthy electrolysis run. The 1.0 M solution was finally made standard for the experiment.When sidelighted, it was transparent enough to permit the observation of electrode surfaces; yet, its concentration was close to that of the full-strength solution and, therefore, readings obtained with 1.0 M solution should be comparable to results of measurements on a full-strength electrolyte.

### E. Cleaning Solutions

An extremely large variety of metal-cleaning formulas is used by the plating industry. Since they all fall into essentially two categories, i.e., alkaline solutions used with or without current, and acid solutions used with or without current, it was not too difficult to select a typical alkaline soak-cleaning and electro-cleaning formula, as well as two acid pickles. For the acid pickles, HCl and $H_2SO_4$ were selected because they are used by almost every plating shop. The use of an acid electro-cleaning bath was considered, but decided against. In many instances, this type of cleaning produces a rather deep etch which improves adhesion but not the appearance of the deposit. Cleaning formulations containing ions of non-alkali metals (such as formula used in the Bullard-Dunn or other strike cleaning processes) were not taken into consideration. These are used cathodically and therefore potential readings would, naturally, be influenced by the type and amount of metal deposited on the cathode while it was being cleaned.

In all cases, including the trial runs, the electrolysis was conducted with low-carbon steel anodes (rather than with lead anodes) since the steel anodes were easier to prepare in such a way that the initial cathode-anode emf was reasonably close to zero. A new anode was used for each cathode sample and the anode area was adjusted to 250 $mm^2$, i.e., about three times that of the apparent area of the cathode.

Sample cathodes used during the experiment were taken at random from a large (several thousand pieces) production lot, immediately after the tempering operation. Physically, samples were small pins, 1.8 mm in diameter, 30 mm long, with one end stamped flat. This shape permitted an easy insertion into the cathode holder, which was provided with a centrally -located 1.9 mm bore and a slot to accomodate the flat end of specimen (see Fig. 4). Pins were manufactured from 1.3% carbon steel and heat treated to a hardness of Rockwell C 60.

The temperature of the electrolysis cell was not thermostatically controlled, but the environment of the experiment was such that the cell temperature remained at 18±1°C during the experiment. The emf for the polarization of samples was obtained from a potentiometer placed across two 6v Eveready dry cells, connected in series. A 6-volt storage battery was used as the current source for electro-cleaning and stripping of samples.

When graphs of the cathode potential (called for in step 15 of the experimental procedure, charts No. 1a through No.4c, showing results of the experiment grouped by the type of cleaning schedule used in sample preparation) were constructed, they proved to be too complicated to be used for evaluation of data. In order to facilitate the comparison between the shapes of all plot curves, data was re-plotted as shown in charts No.5 through No. 12. All these charts contain a reference curve depicting potential readings on wet-scoured samples. This was deemed to be an appropriate criterion, since the original practical problem (see Foreword) was solved by means of a similar method of cleaning.

All charts show that the shape of cathode potential plots varies in a seemingly random manner. The fluctuations in the lower and, possibly, the middle current density region (charts No.1 through No.4, with "a" and "b" subscript) may represent various stages of sample surface conditioning by polarizing current. The difference between individual curves begins to become noticeable in the upper current density region, and especially above the topmost inflection point.

Charts No.5 through No.12 show that in all cases the reference curve No.1 (cleaning schedule No.2, wet-scoured pins) seems to be characterized by the most negative potential. The No.4 curve in chart No.8 (cleaning schedule No.13, specimens cleaned anodically, then pickled in $H_2SO_4$) seems, on the other hand, to be the least negative. Curve No.3 in chart No.5 falls somewhere between these two curves, but is closer to the $H_2SO_4$ than to the reference curve.

To interpret the meaning of the difference in the relative position and shape of the above-described curves, a validity test was conducted. The test also served to check the initial assumption that the shape and the relative position of cathode potential curves will reflect the relative effectiveness of various cleaning schedules and thus might be used as a basis for predicting the quality of subsequent electro-deposits. The test comprised actual plating of samples cleaned in accordance with schedules No.2, No.6, and No.13, and taking thickness measu -

rements on the resultant electro-deposit. The thickness of plate was taken into consideration but its adhesion to the basis metal was not tested.

Results of this test are tabulated in table No.28.The appearance of all samples was approximately the same, except that the samples cleaned in accordance with schedule No.13 were,possibly, somewhat duller that the rest of test piecas. When used in conjunction with charts showing pertinent curves, validity test data provides a definite confirmation of the original assumption. The quality of the deposit seems to be directly correlated with the behavior of corresponding potential-current density curves.

# CONCLUSIONS

As stated in preceding parts of this paper, the purpose of the experiment was to investigate the dependency between specific metal-cleaning methods and a measurable parameter which is influenced by changes on metal surfaces that are caused by cleaning. On the basis of experimental findings, an objective test procedure suitable for evaluation of the relative effectiveness was to be proposed. The results of the experiment, as tabulated and discussed above, warrant the following conclusions.

1. The quality of an electrodeposited coating and the ease of deposition are influenced by methods used in preparing the basis metal for plating. For a specific basis metal, there is a direct correlation between the quality of the deposit and the cleaning method used.

2. The single-electrode potential of the cleaned basis metal is one of the measurable parameters that can be used in evaluating the relative effectiveness of several cleaning methods, or in predicting the quality of the subsequent electrodeposit. The numerical values of this potential become meaningful only when tied in with corresponding densities of polarizing current, viz., when used in plotting potential vs. current density curves. When two individual cleaning methods are being compared , the method which, for a given current density, causes metal to acquire a more negative potential will most likely produce an easier-to-plate surface.

3. When plating chromium over polished and hardened high-carbon steel, the steel surface should preferably be prepared by mechanical cleaning, i.e., by wet-scouring with a mildly abrasive slurry. When mechanical cleaning is not feasible, the metal might be cleaned by degreasing followed by pickling in dilute HCl. In this case, the quality of plating will be comparable to, but not quite as good as, the plating obtained on a mechanically-cleaned surface. The use of $H_2SO_4$ pickle, especially in a final step of cleaning cycle, should be avoided since it is likely to produce an unsatisfactory plate.

Techniques employed in the course of this experiment might be adapted to produce an instrumental method for the evaluation of effectiveness of cleaning procedures. The evaluation would be on a strictly relative basis and, therefore, at least two different cleaning procedures should be tested simultaneously. Such test might be conducted as follows.

1. Obtain several specimen parts from the lot to be plated.
2. Working with one part at a time, clean the surface of the metal using one of the cleaning methods to be evaluated. Immediately after the last cleaning step, make the part the cathode in an electrolyte which is similar to that in the plating tank.
3. Measure single-electrode potential of the specimen at several densities of polarizing current, ranging from zero c.d. to a c.d. only slightly lower than that required for plating.
4. Repeat steps 2. and 3. to obtain multiplicate readings for all cleaning methods. Average each set of multiplicate values.

5. Plot potential vs. current density curves for all cleaning cycles. Visually, compare relative positions of these curves; a curve that is more negative than the others will correspond to the most efficient cleaning cycle.

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