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A polarographic analysis of the tetracyanoaurate (III) and the dicyanoaurate (I) complexes

David Jefferson McClemens
Union College - Schenectady, NY

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**A POLAROGRAPHIC ANALYSIS OF THE
TETRACYANOaurate (III) AND THE DICyanoaurate (I) COMPLEXES**

by

David Jefferson McClemens
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UC 1964

**Senior Thesis Submitted
in Partial Fulfillment
of the Requirements of Graduation**

DEPARTMENT OF CHEMISTRY

UNION COLLEGE

MAY 1964



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1964

This Thesis

Submitted by

Dwight J. McLennan

to the

Department of Chemistry of Union College

in partial fulfillment of the requirements of the degree of

Bachelor of Science with a Major in Chemistry

is approved by

Robert W. Schaefer

TABLE OF CONTENTS

ABSTRACT	v
INTRODUCTION	1
EXPERIMENTAL PROCEDURE	3
DICYANOaurate (I) COMPLEX	6
TETRACYANOaurate (III) COMPLEX	14
SUGGESTIONS FOR FUTURE WORK	28

LIST OF PLATES AND TABLES

		page
Plate I	Apparatus	4
Plate II	Typical Current-Voltage Curve for $\text{Au}(\text{CN})_2^-$	7
Table I	$\text{Au}(\text{CN})_2^-$ Data	8
Plate III	Calibration Curve for $\text{Au}(\text{CN})_2^-$ (I) Data	9
Plate IV	Jacob's Plot for $\text{Au}(\text{CN})_2^-$ (I)	11
Plate V	Current-Voltage Curve for $\text{Au}(\text{CN})_2^-$ (I)	13
Plate VI	Current-Voltage Curve for $\text{Au}(\text{CN})_4^-$ (III)	15
Plate VII	Current-Voltage Curve for $\text{Au}(\text{CN})_4^-$ (III)	18
Table II	Tetracyanoaurate (III) Data	19
Table III	Tetracyanoaurate (III) Data	19
Plate VIII	Diffusion Current vs. Concentration for $\text{Au}(\text{CN})_4^-$ (III)	20
Plate IX	Diffusion Current vs. Concentration for $\text{Au}(\text{CN})_4^-$ (III)	21
Plate X	Change in Diffusion Current vs. Concentration for $\text{Au}(\text{CN})_4^-$ (III)	23
Table IV	Change in Diffusion Current vs. Concentration for $\text{Au}(\text{CN})_4^-$ (III) Data	24
Table V	Jacob's Plot Data for Tetracyanoaurate (III)	24
Plate XI	Jacob's Plot for Tetracyanoaurate (III)	25
Table VI	Jacob's Plot Data for Tetracyanoaurate (III)	26

ABSTRACT

A polarographic analysis of the two gold complexes, dicyanoaurate (I) and tetracyanoaurate (III), was undertaken. Both complexes were shown to have distinguishable current-voltage curves. Previous work on the dicyanoaurate (I) was confirmed, and new data was added for the low concentrations. The tetracyanoaurate (III) was investigated, and the half-wave potential was found to be -0.32 volts. Maxima problems with the complex were encountered and resolved.

INTRODUCTION

Published material on the common cyanide complexes of gold, dicyanoaurate (I) and tetracyanoaurate (III), has been lacking from 1935 until the present. Currently an electron transfer experiment between the two complexes has been anticipated, which requires a simple, quantitative analytical determination of the concentration of the two ions.

After consideration of various methods, the polarographic approach was felt to have much promise since it has the following advantages: (1) only small volumes of the solutions are necessary, (2) the method gives good results at the low concentrations to be used in the experiment, and (3) the process is carried out and computed quickly and easily.

A current versus applied potential curve plot gives a distinct current change for each ion present that is electroreducible. The potential at which this change occurs is called the half-wave potential ($E_{1/2}$). The height of the wave, involving a current change, is proportional to the concentration of the ion species present and is called the diffusion current (i_d). Also, as long as sufficient differences in half-wave potentials exist, two species may be analyzed for, in the presence of one another. Thus, it is evident that this method is capable of performing the necessary job as long as there is sufficient half-wave difference.

The previous published work (2, 5) in this area is rather limited, with only two sources available. John Herman (2) wrote a paper for the Physical Chemical Institute, Prague, Czechoslovakia, published in the Collection of Czechoslovak Chemical Communications, 1934, which points

out that the two complexes are indeed able to be analyzed by polarographic methods. His paper is rather limited in that he gives little or no background data for his measurements, thus making them quite hard to duplicate.

The second, and more comprehensive work, was done by Peter Neddermeyer (5) as part of his degree requirement at Union College. His work was mostly with the dicyanoaurate (I) complex - and was at a higher concentration range (10^{-1} - 10^{-2}) than was necessary for the proposed experiment.

The synthesis of the tetracyanoaurate (III) compound was taken from a thesis by George Hartwell (1).

EXPERIMENTAL PROCEDURE

The main piece of apparatus consisted of the Sargent Model XII polarograph. Other apparatus included the electrode vessel and the dropping mercury electrode. The electrode vessel was constructed in such a manner (See Plate I) as to allow nitrogen to be bubbled through the solution to deaerate it; and also, by the turn of a stopcock, to allow nitrogen to bubble over the top of the solution during the polarogram.

The dropping mercury electrode, d.m.e., itself consisted of a fine capillary and a combination pool and column of mercury above the capillary. The pool and column arrangement was used so that the change in height of the mercury column over the capillary would be almost constant over the period of time necessary for a polarogram to be taken (See Plate I).

The drop time was kept between three and four seconds for all the data contained in this paper. It should be noted that the drop time must be held constant for each polarogram. The drop time should be checked often, and the mercury height adjusted so that the time remains constant. This is important when the following equation is noted (4):

$$i_d = 607 D^{1/2} C m^{2/3} t^{1/6}$$

i_d = diffusion current

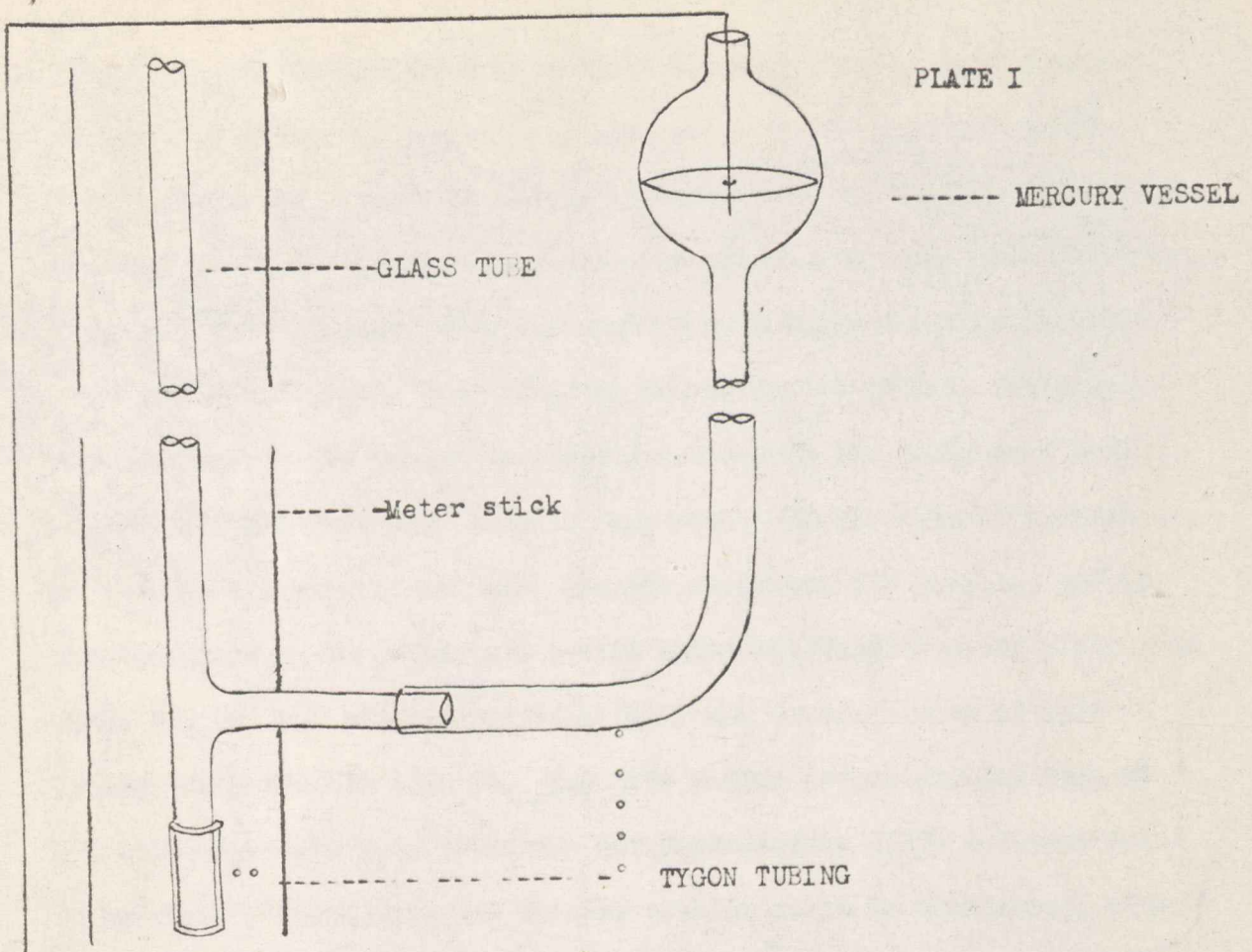
n = number of electrons consumed
in the reaction

D = diffusion coefficient

m = flow (mg/sec) of Hg

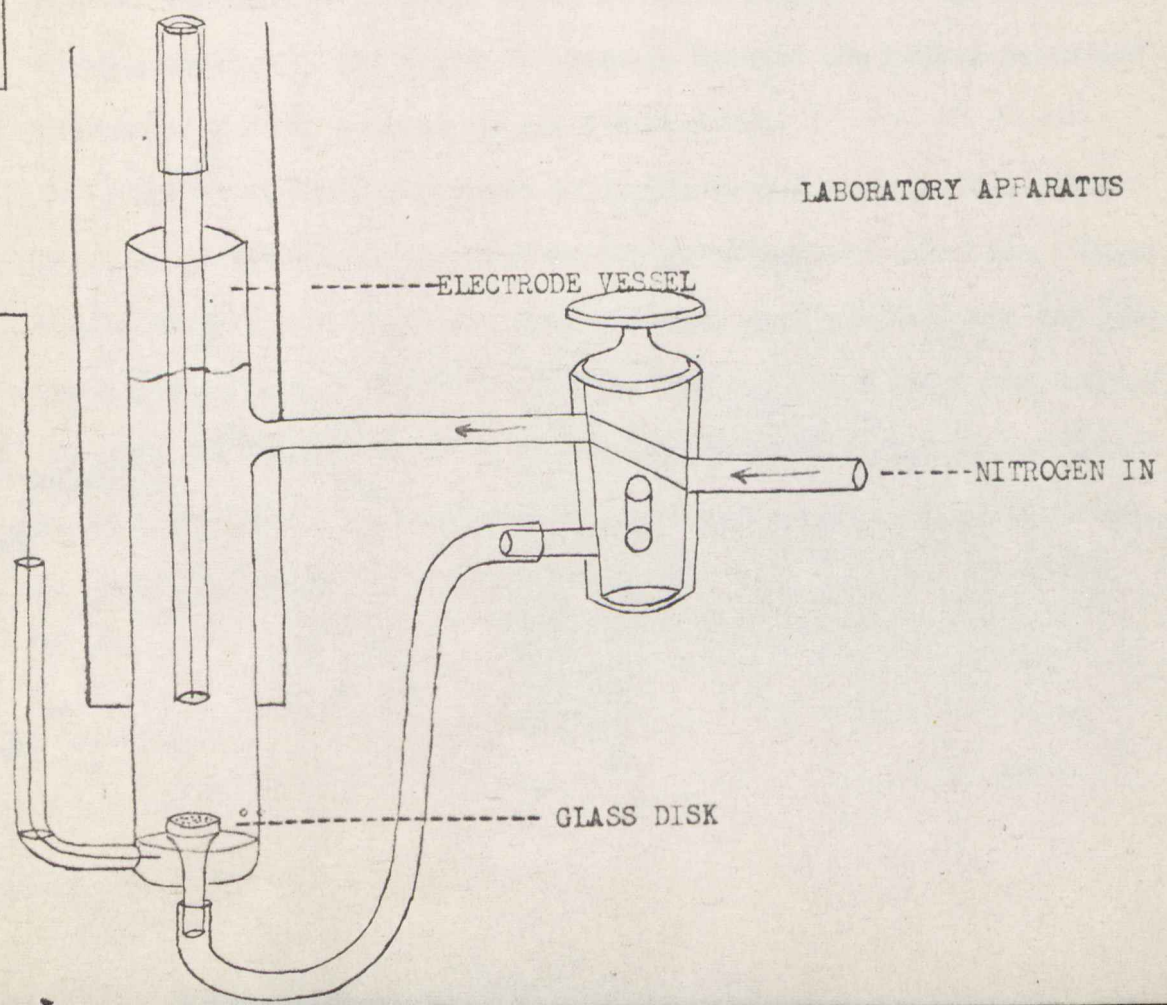
t = drop time

PLATE I



Electrodes
the
larograph

LABORATORY APPARATUS



Thus, it is important to be sure the drop time is kept constant, so that the diffusion current - proportional to t - will not be adversely affected. Also, it should be noted that the capillaries should at least be close to equal in size. The actual drop time used - between three and four seconds - was recommended by Koltoff and Lingane (3).

The solution was then added to the electrode vessel, deaerated, and the mercury was raised to the correct level, the instrument connected, and the data automatically recorded. In the deaerating process, no trouble was experienced with the dicyanoaurate (I) complex, and it was deaerated in the electrode vessel using the fretted glass plate (see Plate I), but the tetracyanoaurate (III) was found to foam if this method was attempted with it. For this reason it was decided that if all solutions used to make up the tetracyanoaurate (III) solution would be previously deaerated, the foaming problem could be eliminated, since further bubbling of nitrogen would be unnecessary. The cause of the foaming problem is not known at present; however the method described eliminated any discernable oxygen interference.

One major source of error in trying to get reproducible polarograms is an incomplete sweeping of the solutions with nitrogen. Water pumped grade of nitrogen was used, and this was purified over hot copper. The tetracyanoaurate (III) complex must be carefully swept, or its components, for a large peak due to gaseous impurities will be encountered in the area of the complex's half-wave region, thus making it difficult to analyze the wave.

DICYANOaurate (I) COMPLEX

The dicyanoaurate (I) complex was investigated for two reasons, (1) to confirm the results of Neddermeyer, and (2) to work at lower concentrations, so that the concentrations which would be used in the actual electron transfer experiment would have been investigated.

The dicyanoaurate (I) complex is commercially available and any desired stock solution could be made up and aliquots thereof could be used. The supporting electrolyte used was potassium cyanide. This selection was made so as to keep the number of ions in solution in the solution at a minimum. Also important is the fact that the cyanide complexes of gold are quite stable, since the cyanide can contribute electrons as a ligand and form pi bonds with the gold, and thus make the complex more stable.

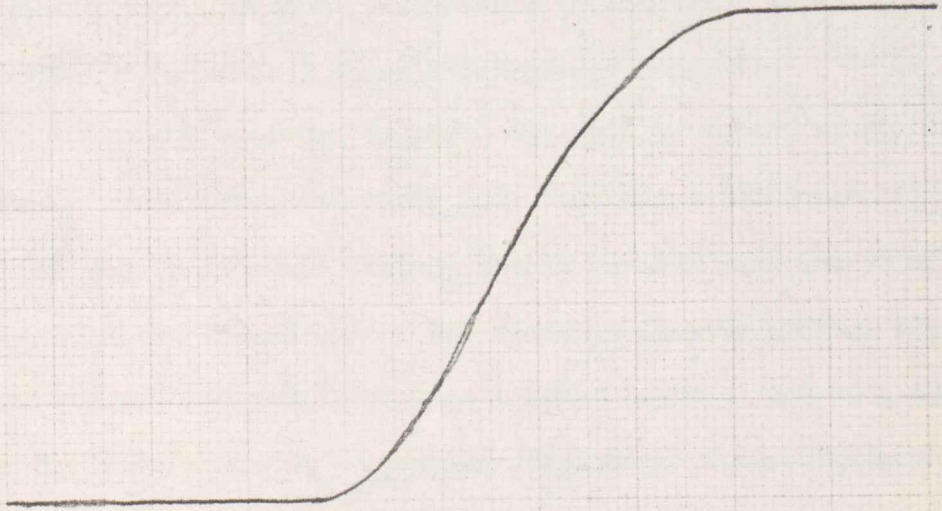
Also, the method of analysis of the current-voltage curve should be mentioned. A particular point on each of the two plateaus, the limiting and the residual, was picked, and all measurements were taken from these points. It doesn't matter which points are used, but they must be kept constant, thus giving equal weight to each measurement.

The results of the analysis showed a half-wave potential of -1.32 volts, quite close to the value that Neddermeyer obtained, -1.30 volts (5), (See plate II for a typical curve.) The slopes of the calibration curves, diffusion current versus concentration, were slightly different, however. Neddermeyer's value for this plot had a slope of 2.286 milli-amp/mole/liter, while the data recieved for various polarograms at the same and lower concentrations was 4.26 milli-amp/mole/liter (See Plate III), (5). There was a slight change in concentration of the supporting

TYPICAL CURRENT-VOLTAGE CURVE
FOR $\text{Au}(\text{CN})_2^-$

CURRENT

APPLIED VOLTAGE



electrolyte in the later polarograms, so that its supporting electrolyte would have the same concentration as the tetracyanoaurate (III). The tetracyanoaurate (III) was using a lower concentration for reasons which will be discussed later, but it was felt that the two should have the same concentration supporting electrolytes as they will in the electron transfer experiment, when they are polarographed together.

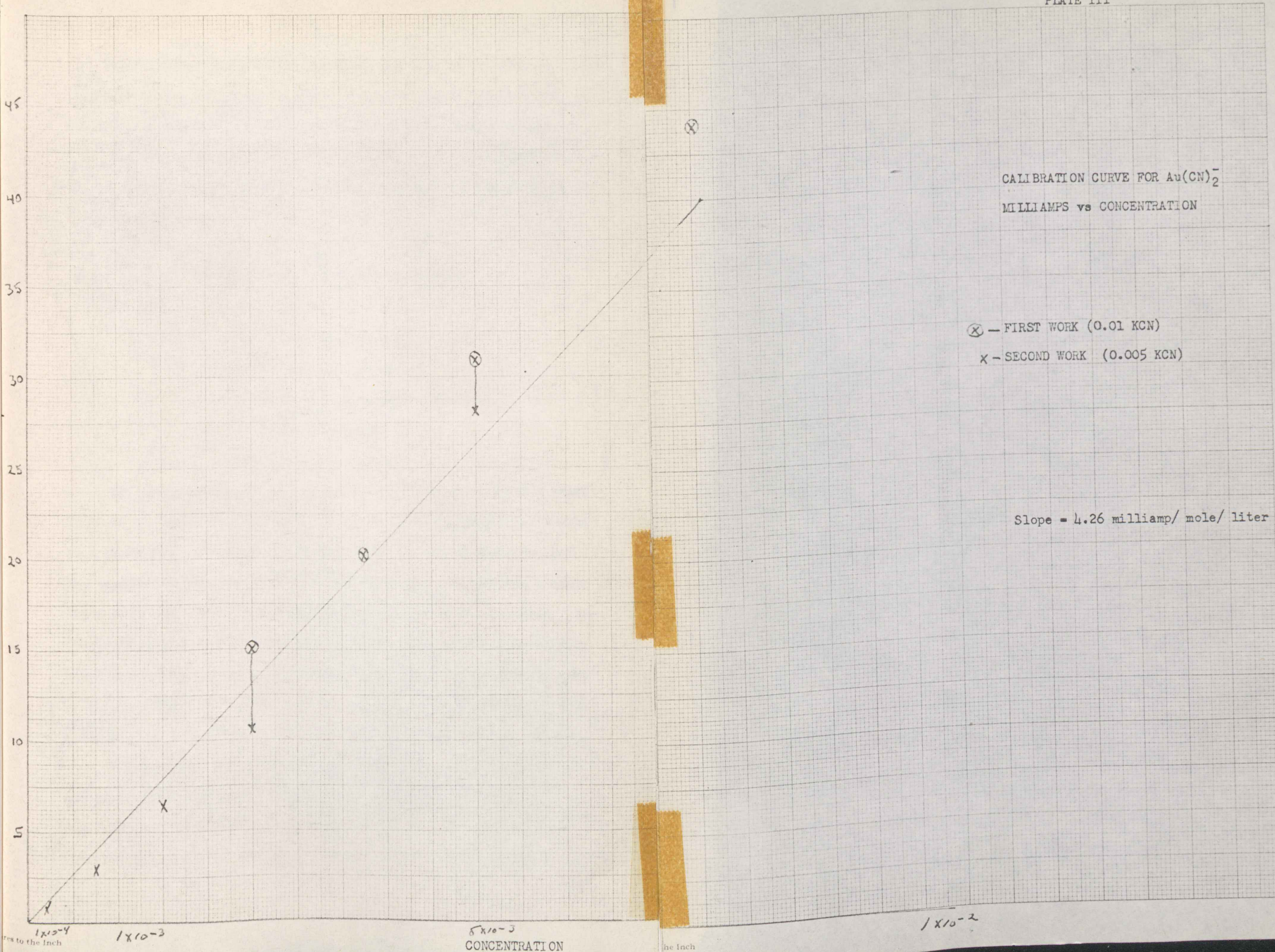
This change may account, in part, for the low values of the low concentrations on the graph on Plate III. There is a difference in the slopes of the two calibration curves, but it is felt that the difference can be accounted for in two ways. The first is that in neither case was a good, mathematical approach, such as a least squares analysis, used to determine the line. The low supporting electrolyte concentrations may also contribute to the difference.

Below are some typical data, from which Plate III was drawn:

TABLE I

$\text{Au}(\text{CN})_2^-$	KCN	i_d
7.5×10^{-3}	1×10^{-2}	44.1 $\mu\text{a.}$
5.0×10^{-3}	1×10^{-2}	30.9 $\mu\text{a.}$
5.0×10^{-3}	5×10^{-3}	28.0 $\mu\text{a.}$
3.75×10^{-3}	1×10^{-2}	20.2 $\mu\text{a.}$
2.5×10^{-3}	1×10^{-2}	15.1 $\mu\text{a.}$
2.5×10^{-3}	5×10^{-3}	10.3 $\mu\text{a.}$
1.25×10^{-3}	5×10^{-3}	6.15 $\mu\text{a.}$
6.25×10^{-4}	5×10^{-3}	3.1 $\mu\text{a.}$
1.25×10^{-4}	5×10^{-3}	0.75 $\mu\text{a.}$

(It should be noted that the above data does not include simply results from just one polarogram, but is a composite of various polarograms.)



There was no trouble with maxima in the case of the dicyanoaurate (I). The concentration of the supporting electrolyte was kept low, at the level of 5×10^{-3} , to be consistent with future experimentation.

The dicyanoaurate (I) was also examined to see if the reaction was reversible. To do this, the Nernst equation, or a modification thereof, was used (4):

$$E_{d.e.} = E_{\frac{1}{2}} - \frac{0.0591}{n} \log \frac{i}{i_d - i}$$

$E_{d.e.}$ = potential of d.m.e.

$E_{\frac{1}{2}}$ = half-wave potential

n = number of electrons involved

i = current height

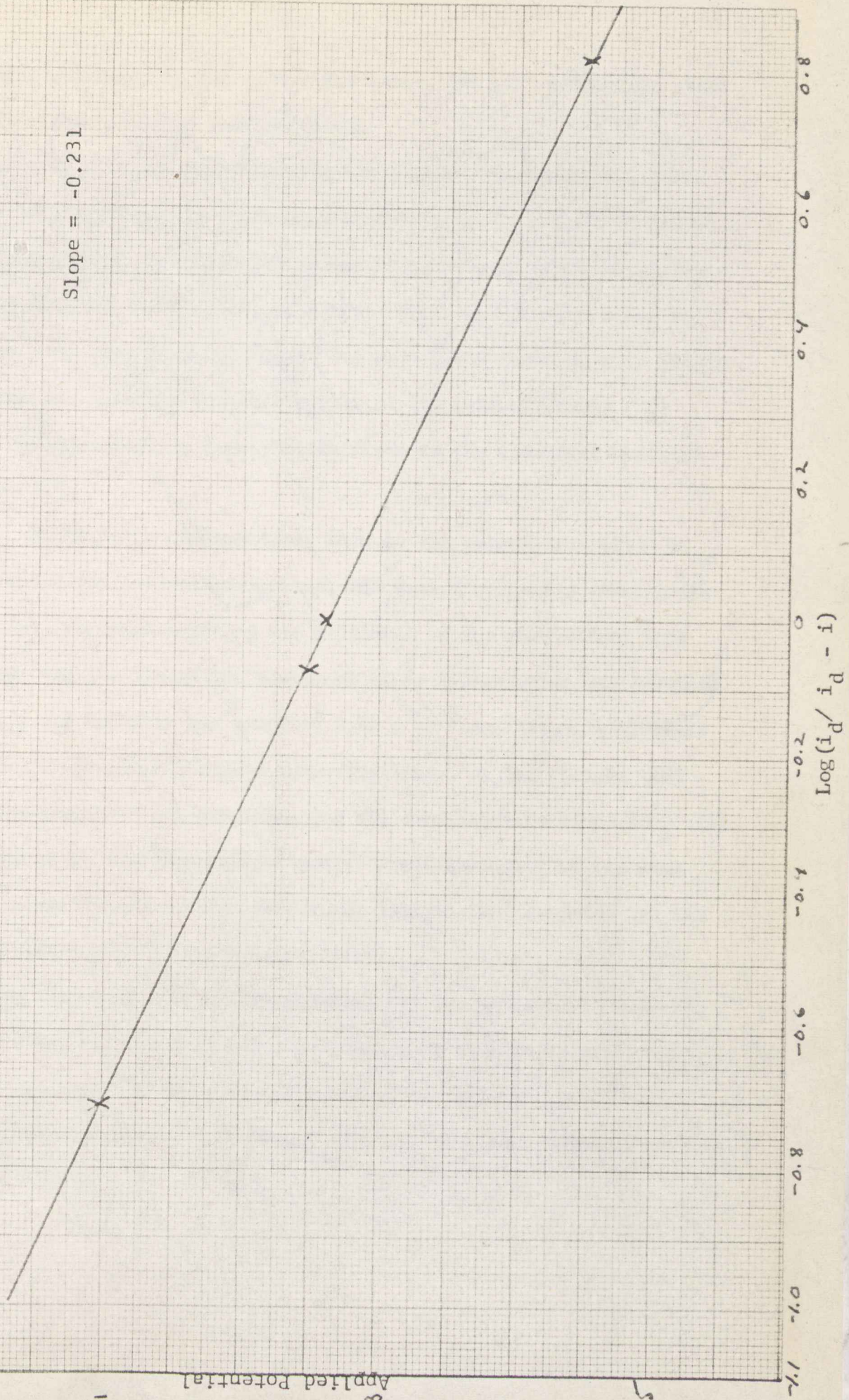
i_d = diffusion current

If, then, a plot of $E_{d.e.}$ vs. $\log i/(i_d - i)$, or a Jacob's Plot, is made, the slope of this line, if the reaction is reversible, should be $- (0.059/n)$. An example of such a plot is shown on Plate IV, and the value of -0.231 was arrived at for the slope. Calculating a value for n from this slope, one finds the answer to be 0.255 electrons. As mentioned in Meites and Thomas (4), an error of $\pm 10\%$ is conceivable for a reversible reaction. Even within this error, the value arrived at does not approach a whole number, which it would have to be if the reaction were reversible. This is in agreement with earlier work (5).

The equation also offers an opportunity for calculation of a precise theoretical value of the half-wave potential. If the term $\log i/(i_d - i)$ is equal to zero, then the $E_{d.e.}$ will equal the $E_{\frac{1}{2}}$. When computed from the graph, this value equals 1.27 volts, which is

POTENTIAL versus Log ($i_d / i_d - i$)

Slope = -0.231



just slightly lower than the calculated value, but well within the range of the experimental error involved here.

New work with the dicyanoaurate (I) complex was undertaken when it was discovered that the ion-exchange problem would be carried out at low concentrations. As Plate III demonstrates, the data for these low concentrations fit the previous data very well. One problem which was encountered was the disappearance of the wave after three or more polarograms were run from a particular sample at low concentrations. (A "sample" is approximately twenty milliliters in the electrode vessel.) (See Plate V.)

For the higher concentrations, five or six polarograms could be taken, but for the low concentrations, the wave disappeared after a few polarograms. Constant stirring was initiated to see what effect this would have, but the phenomenon continued after the solution was stirred. This effect was noted at the level of 1.25×10^{-3} and lower, with indications of it at slightly higher concentrations. It was thought that perhaps the reduction was complete, and the concentration of $\text{Au}(\text{CN})_2^-$ (I) had dropped below the discernable level. Since the reaction has been shown to be irreversible, this idea seems logical, and explains why the phenomena occurs only at low concentrations.

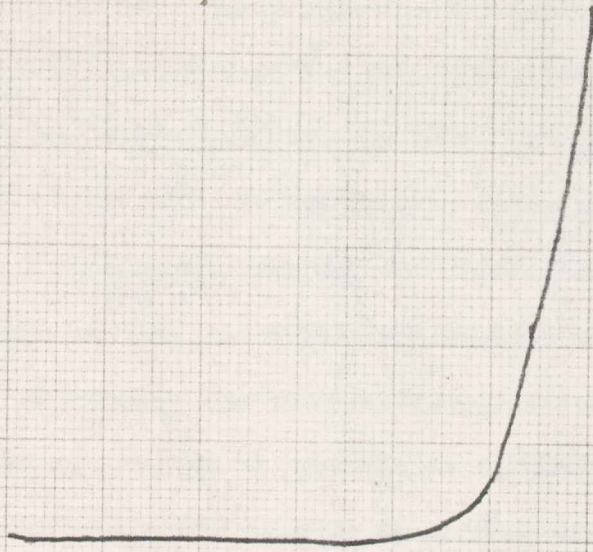
Aside from this difficulty, however, the low concentrations were little trouble, and the data was consistent with that found previously. The first run data was found at all times to be reproducible, thus indicating that duplicate polarograms will not be a necessity for the experimental work.

EXCESS POLAROGRAPH

LOW CONCENTRATION $\text{Au}(\text{CN})_2^-$

CURRENT

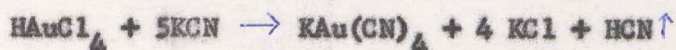
APPLIED VOLTAGE



TETRACYANOaurate (III) COMPLEX

Previous examination of this complex was rather limited (2,5), the only reports stating that the polarographic method was useful in analyzing the material, and that the half-wave potential was approximately - 0.3 volts. (5). It remained to investigate the current-voltage plots of this complex, correct any difficulties interfering with analysis of the curves, and supply calibration curves.

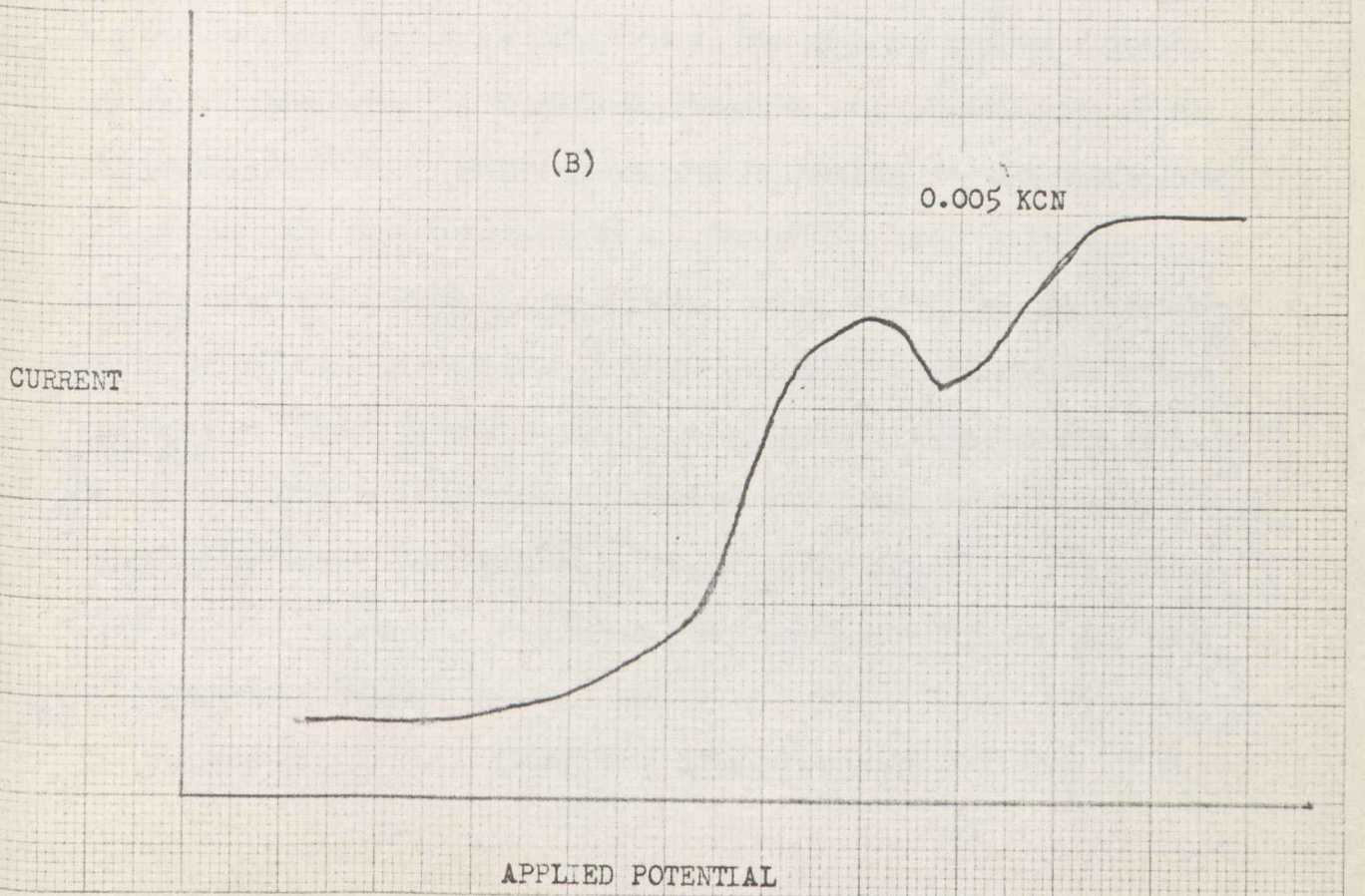
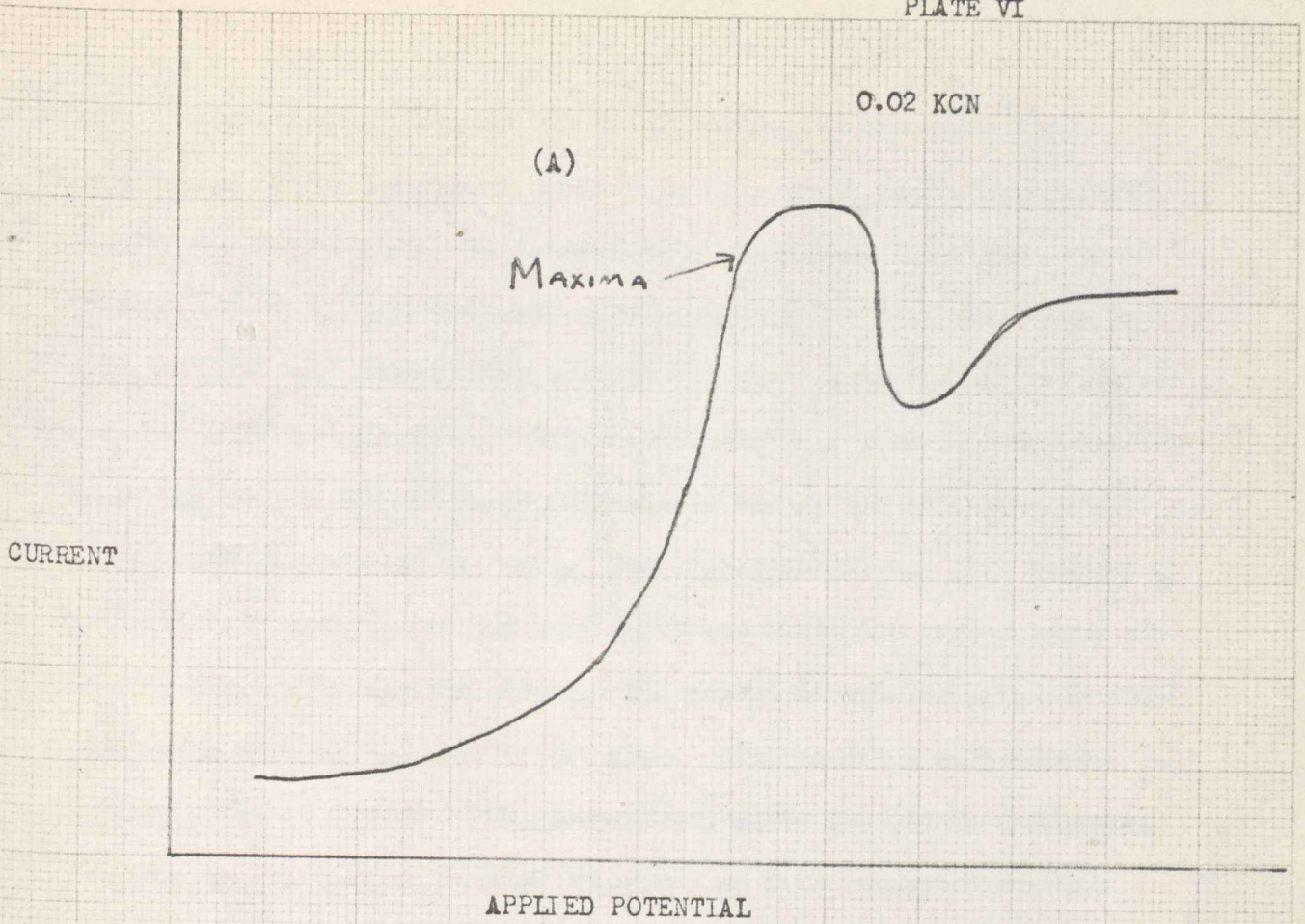
Before discussing the current-voltage curves, however, it is pertinent that the method of preparation of the complex be mentioned. The tetracyanoaurate (III) complex of gold is not commercially available and must be synthesized when needed. George Hartwell's thesis was devoted to the subject (1) and he recommended the following method:



The directions for this reaction must be carried out painstakingly or the product will be affected.

The half-wave curve for this complex (See Plate VI A) is not quite as simple to analyze as is the wave for the dicyanoaurate (I) complex. It gives no clear half-wave, thus causing much thought about how to analyze the wave. The unclear portion of the wave is called a polarographic maxima, and makes it difficult to see which plateau is the limiting one. The maxima is usually caused by high ionic strength of the solution or by some interfering species which is being reduced at the same potential as the particular ion being investigated. An attempt was made to eliminate this hindrance before actual analysis of the complex was undertaken.

There are two possibilities which come to mind in reference to



this problem, the variance of the concentration of the supporting electrolyte, and the variance of the pH. The first to be investigated was the concentration of the supporting electrolyte. Previous polarograms had been made at a concentration of 1×10^{-2} M KCN, and therefore the problem was attacked using this as a base. First the concentration of the potassium cyanide was raised to 5×10^{-2} , 1×10^{-1} , and 5×10^{-1} . This only exaggerated the maxima, however, and at the highest concentration almost obliterated the wave. The concentration was then lowered to 7.5×10^{-3} , 5×10^{-3} , and 2.5×10^{-3} . These dilutions gave a curve similar to Plate VIB, which eliminates the maxima to some extent, but still gives more than one plateau to the plot. (It was found that after a concentration of 5×10^{-3} KCN was reached, there was little difference noted in the plots with further dilution, so this concentration was chosen as the best for the supporting electrolyte.)

The pH change was also investigated. At the concentration of 5×10^{-3} for the supporting electrolyte, the pH for a typical solution was 10.4. This value was largely dependent on the concentration of the supporting electrolyte, and thus was fairly constant for all polarograms of the different concentrations of the tetracyanoaurate (III) ion. First, the pH was raised, in graduations, up to 11, through the addition of sodium hydroxide. Then it was lowered to 9.8, by addition of hydrochloric acid. Both of these changes, although affecting the size and shape, were of no visible value in eliminating the maxima. It might be noted at this point that Neddermeyer found similar results with the dicyanoaurate (I) complex, so that the two complexes have similar reactions to the various changes (5).

Having made these observations, it is interesting to make

conjecture as to why the change in strength of the supporting electrolyte was able to reduce the maxima. The work of Neddermeyer shows the cause of the maxima in the dicyanoaurate (I) complex to be an increasing ionic strength of the solution, caused by a higher concentration of the supporting electrolyte. He states that the higher ionic strength causes the dropping mercury electrode to momentarily depolarize, thus causing the maxima, (5). This seems to be the solution in the present case, too, for the circumstances are much the same.

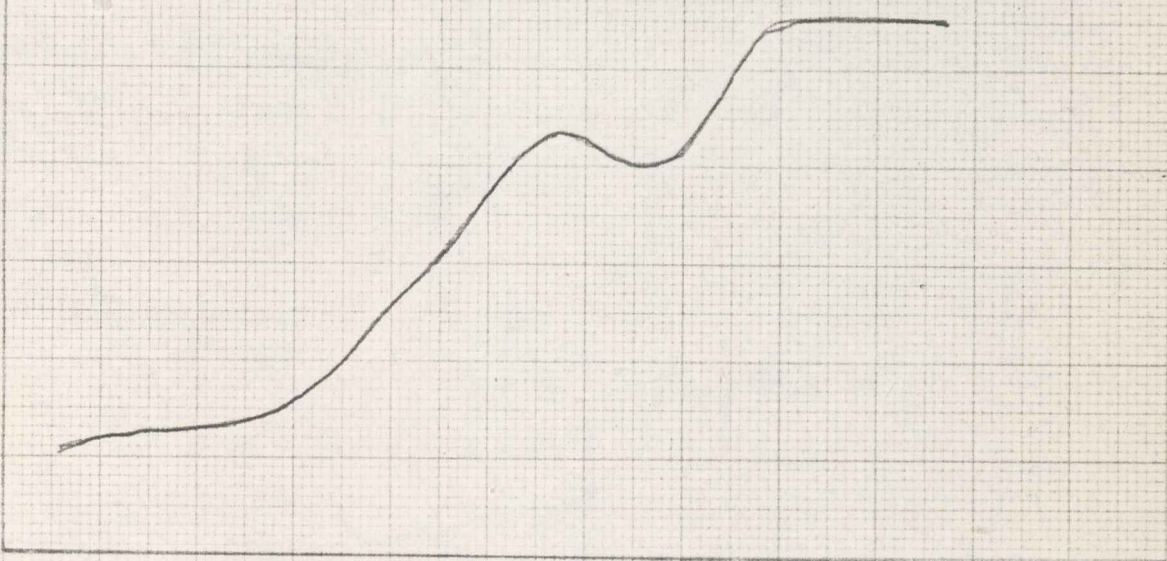
It is to be noted that the concentration of the supporting electrolyte, for the higher concentrations of the complex, i.e., 5×10^{-3} and higher, is not at the recommended value of fifty to one hundred times higher than the ion species being analyzed, and this would be a deterrent if the electron transfer experiment were to be carried out at high concentrations (4). This fact should be remembered if high concentration polarograms are taken.

After attempting to eliminate the maxima in the aforementioned manner, the addition of a wetting agent, Triton X-102, was considered. If the ionic strength of the supporting electrolyte was too high even at this low level (5×10^{-3}) of concentration, perhaps the addition of the Triton X-102 would work to combat this deficiency of the d.m.e.

Adding a few drops of a dilute solution of the wetting agent caused interesting results (See Plate VII). However, there was not much duplication in this area. The Triton X-102 enabled the current-voltage curve to be read with much more assurance, since it eliminated one of the plateaus, and made it clear that the main residual plateau was in the neighborhood of 0.2 volts, and the limiting plateau was -0.8 volts. Thus, these two places were used to evaluate all the

CURRENT

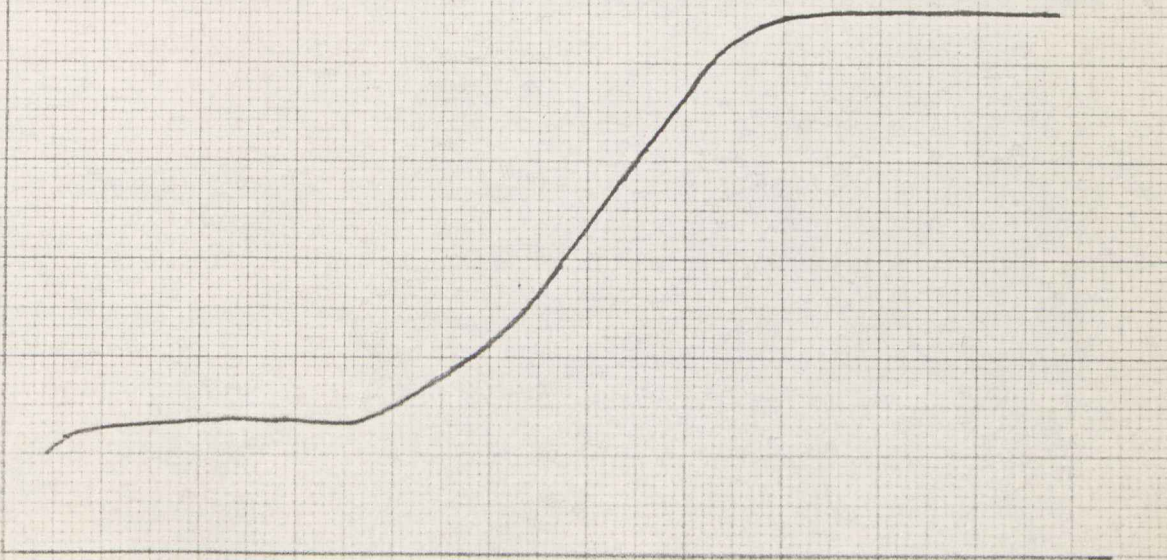
$1.87 \times 10^{-3} \text{ Au(CN)}_4^-$
 $5 \times 10^{-3} \text{ KCN}$



APPLIED POTENTIAL

CURRENT

$1.87 \times 10^{-3} \text{ Au(CN)}_4^-$
 $5 \times 10^{-3} \text{ KCN}$
Triton X-102



APPLIED POTENTIAL

tetracyanoaurate (III) current - voltage curves.

Having discovered how to best read the recorded data, the current-voltage curves for the tetracyanoaurate (III) could be analyzed. A calibration line for the high concentration is plotted on Plate VIII, and is from the following data:

TABLE II

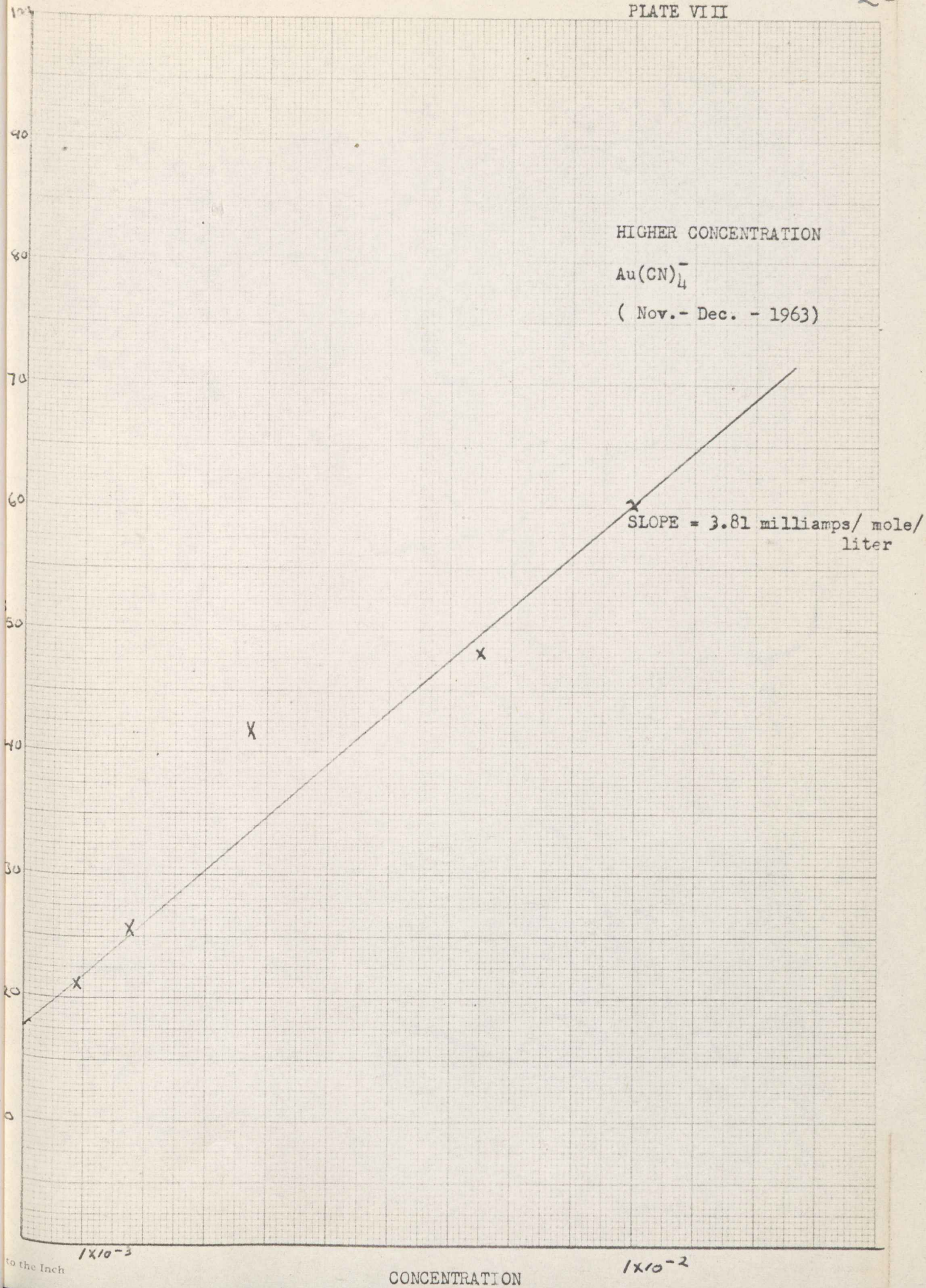
Au(CN)_4^- conc.	KCN	$\frac{i_d}{d}$
1.0×10^{-2}	5×10^{-3}	22.5
7.5×10^{-3}	5×10^{-3}	11.1
3.75×10^{-3}	5×10^{-3}	4.5
1.88×10^{-3}	5×10^{-3}	2.75
9.9×10^{-4}	5×10^{-3}	1.2

The slope of the calibration curve was 3.81 milli-amp/mole/liter.

The data from a lower set of concentrations for the tetracyanoaurate (III) complex, the range in which the electron transfer experiment will be carried out, is plotted on Plate IX, from the following data:

TABLE III

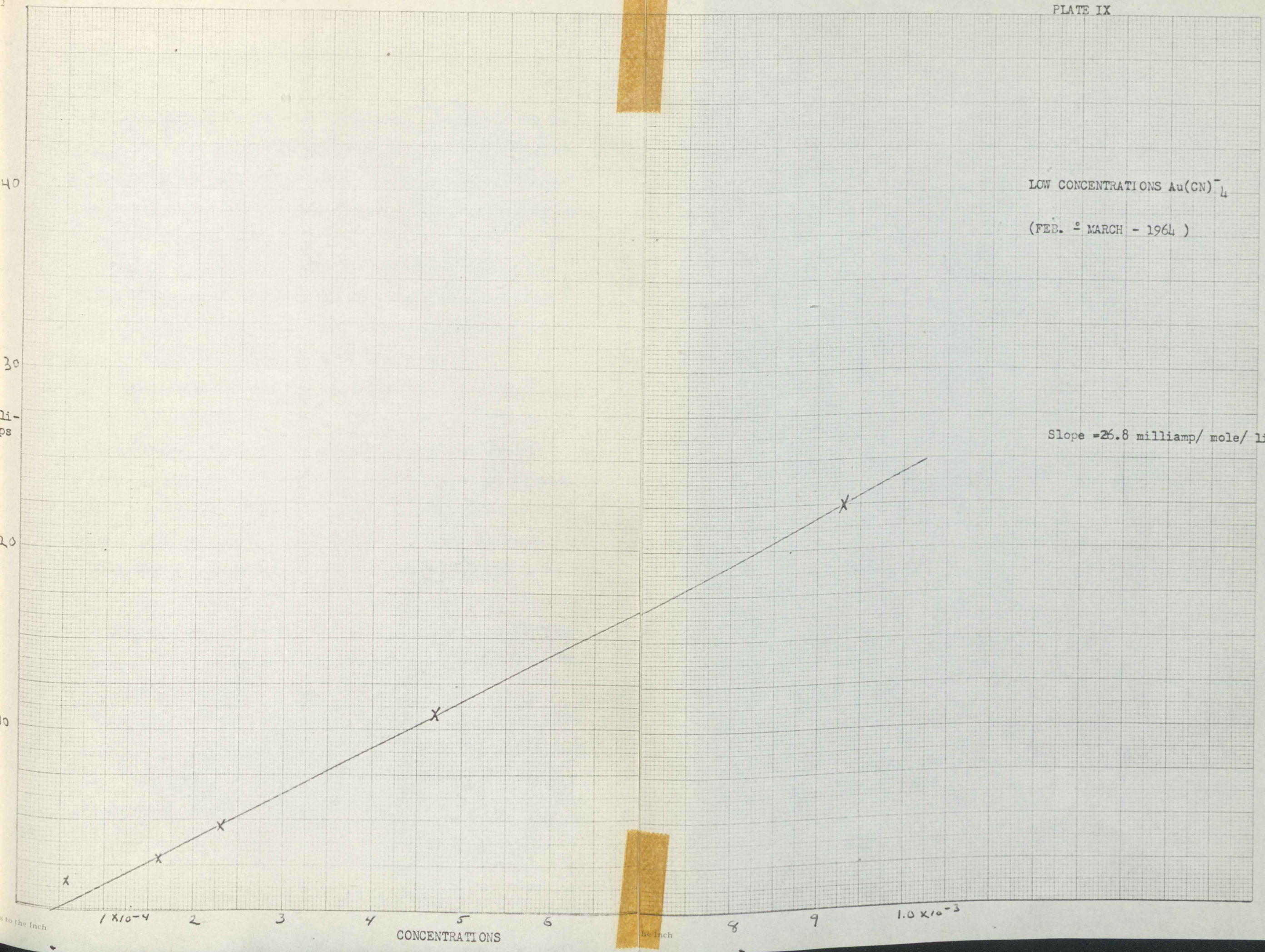
Au(CN)_4^- conc.	KCN	$\frac{i_d}{d}$
9.3×10^{-4}	5×10^{-3}	22.5
4.65×10^{-4}	5×10^{-3}	11.1
2.33×10^{-4}	5×10^{-3}	4.5
1.60×10^{-4}	5×10^{-3}	2.75
5.80×10^{-5}	5×10^{-3}	1.20



LOW CONCENTRATIONS $\text{Au}(\text{CN})_4^-$

(FEB. - MARCH - 1964)

Slope = 26.8 milliamp/ mole/ liter



The slope of the calibration curve for this line is 26.8 milli-amps/mole/liter.

Obviously there is something wrong, since the slopes of the two calibration curves of the tetracyanoaurate (III) complex do not agree and they do not go through the origin - although the low concentration is reasonably close. This immediately casts doubts as to the accuracy of the method of analysis used.

However, when the data is examined, each of these curves was found to be constructed from data made from different runs of the synthesis of tetracyanoaurate (III). This implies that there could be a source of error in the production of the complex itself.

Looking closely at the method of synthesis, it is recognized that there is the possibility of impurity. The synthesis is most exacting as to volume requirements, and the constant stirring method used to eliminate the HCN leaves doubt as to the completeness of the reaction. (Yields of 45-50% are common.) Also, due to the high cost of the chlorauric acid, only one recrystallization of the salt was performed, purity suffering. The yield at all times equaled or exceeded Harwell's, so that it is felt that the method was carried to completion correctly.

Assuming that part of the reason that the calibration curves fail to have the same slope and do not run through the origin may be due to this process, how may this be shown?

If a plot of change in diffusion current versus change in concentrations of the tetracyanoaurate (III) complex is made, and if this line is through the origin, this indicates that the original concentrations weren't correct. Plate X, plotted from the following data from Plate IX, shows this to be the case:

CHANGE IN DIFFUSION CURRENT
VS
CHANGE IN CONCENTRATION
(FROM PLATE IX)

15

10

5

1

1 2 3 4 5 6
 Δ CONCENTRATION ($\times 10^{-4}$)

Centimeters to the Inch

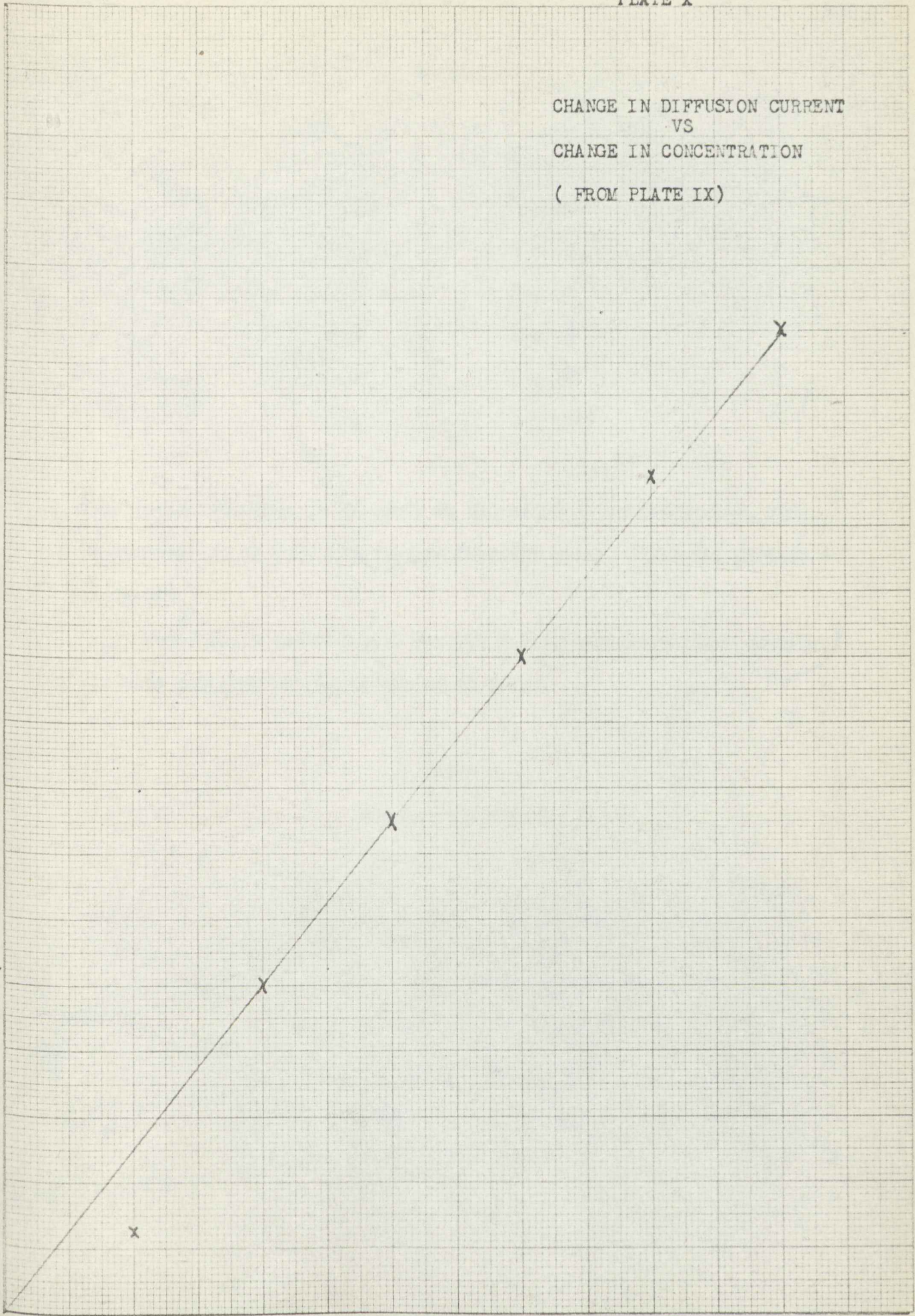


TABLE IV

Δi_d	Δ Concentrations
1.25	1×10^{-4}
5	2×10^{-4}
7.5	3×10^{-4}
10.0	4×10^{-4}
12.75	5×10^{-4}
15.0	6×10^{-4}

This indicates there is an error in the original concentration, thus implying there is some irreducible impurity present from the process of synthesis.

Plate XI, a Jacob's plot for the tetracyanoaurate (III) complex, was made from the following tables of data:

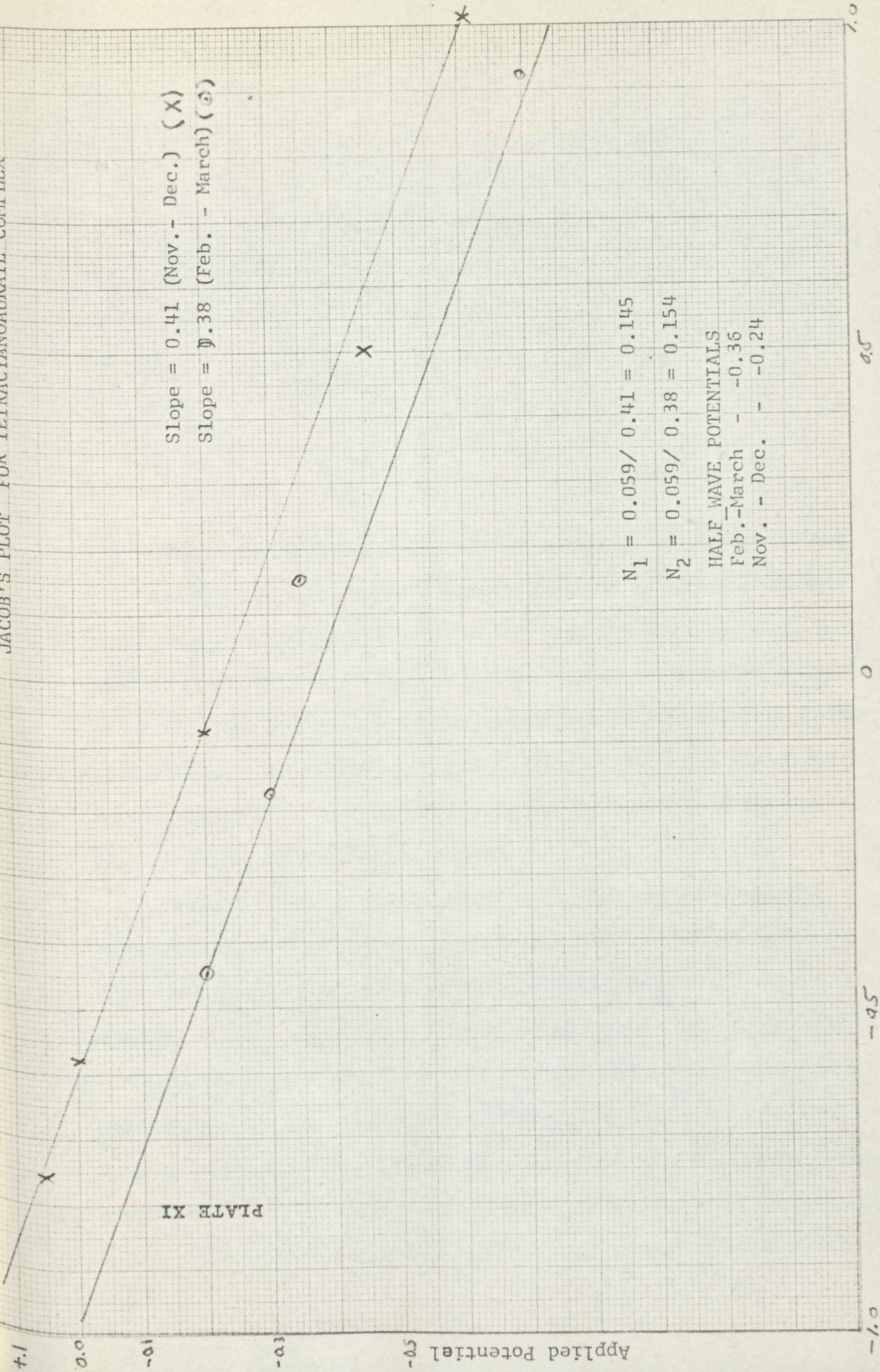
TABLE V

(February - March)

Applied Potential	$\text{Log } 1/(i_d - 1)$
0.0	1.4
-0.20	-0.44
-0.30	-0.17
-0.45	+0.17
-0.70	+0.92

JACOB'S PLOT FOR TETRACYANOAUATE COMPLEX

PLATE XI



Slope = 0.41 (Nov. - Dec.) (x)
 Slope = 0.38 (Feb. - March) (o)

$N_1 = 0.059 / 0.41 = 0.145$
 $N_2 = 0.059 / 0.38 = 0.154$

HALF WAVE POTENTIALS
 Feb. - March - -0.36
 Nov. - Dec. - -0.24

0.5

0

-0.5

Log (i/i_D - i)

1.0

TABLE VI

(November - December)

<u>Applied Potential</u>	<u>Log $i/(i_d - i)$</u>
+0.05	-0.764
0.0	-0.582
-0.2	-0.085
-0.45	+0.491
-0.60	+1.02

This plate shows the slopes of both examples, and indicates that this reaction is irreversible. However, again it is demonstrated that there is a discrepancy by noting the fact that for the first plot, the half-wave potential is -0.24, and for the second, the half-wave potential is -0.36. This indicates something irreducible is present, thus shifting the half-wave. As for the slope value, and thus "n" - the slopes are easily equal within experimental error, and the validity of the statement of irreversibility is not doubted.

There is, however, some serious doubt as to the purity of the tetracyanoaurate (III) complex on which the whole experiment is based. This would explain some of the aforementioned phenomena. Another point to consider is that the two sets of polarograms were taken with different concentrations of supporting electrolyte, thus changing the ionic strength of the solutions and shifting the half-wave potential. A third factor that might contribute is the low ion to supporting electrolyte ratio for the higher concentrations of the complex. As mentioned before, this is recommended to be fifty to one hundred times as great,

at least, and it is not in this case.

Possibilities exist that impurities, such as $\text{Au}(\text{CN})_3\text{Cl}^-$, which would be reducible and thus shift the half-wave potential, also could be present and cause some of the difficulties. The later work (February - March) is probably the more accurate as it was carried out at lower concentrations of supporting electrolyte, and it used tetracyanoaurate (III) complex, which was made after the synthesis was perfected to a greater extent.

Thus, the tetracyanoaurate (III) complex has been shown to be quite readily analyzed by polarographic methods. It has a half-wave potential of circa -0.35 volts. This means that a combination of the dicyanoaurate (I) complex and the tetracyanoaurate (III) complex should be able to be polarogrammed without any interference between the two half-wave potentials. Many of the problems of the maxima have been overcome and suggestions have been made as to the causes of the remaining trouble spots.

SUGGESTIONS FOR FUTURE WORK

The usefulness of Triton X-102, or of X-100 if available, needs to be studied to a greater degree. The exact amount necessary should be determined, and more tests should be made.

A more important area of inquiry is the synthesis of the tetracyanoaurate (III) complex itself. Obviously, doubt has been cast as to the purity of the product of this synthesis, and a detailed analysis of the product should be undertaken, so that steps to improve the purity can be made.

The area of the combined polarogram will then be ready to be investigated, and the hypothesis that the two complexes can be analyzed for in the presence of each other can be tested. After this has been accomplished, a major contribution to the chemistry of the complexes of gold will have been made.

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