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SEMI-ANNUAL REPORT

to

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

for

CHARACTERIZATION OF THE PHYSICO-CHEMICAL PROPERTIES
OF POLYMERIC MATERIALS FOR AEROSPACE FLIGHT

NSG-5009

Bowie State College
Bowie, Maryland 20715

(NASA-CR-160070) CHARACTERIZATION OF THE
PHYSICO-CHEMICAL PROPERTIES OF POLYMERIC
MATERIALS FOR AEROSPACE FLIGHT Semiaannual
Report (Bowie State Coll., Md.) 68 p
HC A04/MF A01

N81-31362

Unclassified
CSCL 11G G3/27 32768

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September 2, 1980

A B S T R A C T

The differential thermal analyzer is a very suitable instrument for the rapid analytical study of the thermal behavior of battery electrodes. Solid samples can be studied in the range of 0°C - 500°C using the standard cell assembly. Thermal behavior of battery electrodes is automatically recorded by the analyzer and it can be used for qualitative analysis. A study is also being made of the behavior of battery electrodes which have been charged at different levels.

INTRODUCTION

Differential thermal analyses are conducted with a DuPont Model 900 DTA unit. DTA is a technique for studying the thermal behavior of materials as they undergo physical and chemical changes during heating and cooling. Two 4mm-diameter tubes, one containing sample and the other containing a reference material, such as glass beads, are heated at a uniform rate in a heating block. The temperature differential between the two tubes will remain zero as they are heated unless the sample undergoes an endothermic or exothermic reaction. A thermocouple is inserted in the tube containing the sample and another thermocouple is inserted in the tube containing the glass beads. The glass beads do not undergo any chemical change in the temperature range under study. As long as the temperature of the sample equals the temperature of reference material, the two thermocouples produce identical voltage and the net voltage differential is zero. When an exothermic or an endothermic change takes place in the sample, the sample temperature no longer equals the reference temperature and the resultant voltage differential reflects the difference in temperature and either a positive or negative ΔT peak on the graph results. The DTA unit plots the temperature of the heating block on the X-axis; on the Y-axis it plots the difference in temperature between the sample and the reference, ΔT . An exotherm is plotted as a rise from the base line; an endotherm as a decrease from the base line.

DISCUSSION OF THE RESULTS

Several positive and negative battery electrodes were analyzed. The negative plates show a first endotherm between 245°C - 250°C. This is a very large peak. The second endotherm occurs at 300°C which is indicative of the decomposition of Cd(OH)₂ (see graphs 1 to 6). In the analysis of positive plates, a first weak endotherm occurs at 100°C, which indicates loss of H₂O from Ni(OH)₂(H₂O)_n molecules. A second, large, endotherm occurs in the range of 290°C - 300°C, which is indicative of the decomposition of Ni(OH)₂ to NiO and H₂O (see graphs 7 to 17).

ABSTRACT

Atomic Absorption Spectroscopy is used to determine nickel, cobalt, cadmium, and potassium content in battery electrolytes and electrodes. We are also determining the interference effects of one element in the presence of others. Atomic Absorption is a quick and accurate method for the determination of traces of the above mentioned metals.

Introduction

Sealed Ni-Cd cells have proved to be a useful and reliable rechargeable source of power for aerospace applications. However, it has been found that sometimes these cells have failed.

Although it is not completely known what leads to such failures, it has been found experimentally that some of the factors which contribute to the final failure of the batteries are :

1. Extent and nature of cycle regime
2. Operating temperature
3. Carbonate contamination
4. Cd migration
5. Nature and condition of separator

The analysis of negative electrodes, positive electrodes, and of the electrolyte is also important.

A.A spectroscopy is being used to analyze the elements of interest (Ni, Cd, Co, and K) in the electrodes and electrolytes of the Ni-Cd cells.

These results have been compared with those obtained by standard chemical analysis method and are in agreement. A.A spectroscopy is much quicker and embraces virtually all alloying components contained in Ni-Cd cells.

This method is being used to analyze for concentration of trace metals in negative and positive electrodes of batteries. This should prove useful in determining the amount and effects of these trace metals in functioning and durability of Ni - Cd cells.

Instrumentation

A Perkin-Elmer Model 403 Atomic Absorption Spectrophotometer was used. This unit has a digital read-out panel. High intensity cathode tubes for Ni, Cd, and Co. were used depending on which element was being measured. Operating conditions were generally those recommended in the Analytical Methods Book.

The steps listed below were followed in adjusting the Model 403 Spectrophotometer in preparation for performing the analysis.

1. The instrument and exhaust hood are turned on and allowed warm-up at the specified current given in the Analytical Method Book for two hours or until stability is achieved. Stability is achieved when no zero shift is apparent over a five minute interval.
2. The air supply is turned on and the air pressure is set at 62 lbs./sq. in.
3. The acetylene supply is turned on and acetylene pressure is set at 27 lbs./sq. in.
4. The burner is ignited.
5. The flame should be blue and transparent with an oxidizing region about 4 mm.
6. The slit control is set at the value given in the Analytical Methods Book for the respective elements.
7. The adjustment of the atomizer is made by turning the capillary outward until "blow-back" occurs, then, turning inward until absorption is maximized. Standard solutions are aspirated through a tube into the flame for not less than 15 seconds.

Known Solutions Preparation

The solutions used were prepared from standard solutions of 1000 (Parts per Million (PPM)). The dilutions were made as follows:

10 ml of 1000 PPM standard solutions were diluted to a final volume of 500 ml with deionized water to give a solution of 20 PPM concentration. This 20 PPM solution was used as a stock solution. Further dilutions were made as follows:

1. 5 ml of 20 PPM solution was diluted with deionized water to give a final volume of 100 ml and a solution of 1 PPM.
2. 10 ml of 20 PPM solution was diluted to a final volume of 100 ml and a solution of 2 PPM.
3. Repeat the above procedure with 15 ml of stock solution to get 3 PPM solution.
4. Repeat above procedure with 20 ml stock solution to yield a solution of 4 PPM.
5. Repeat above procedure with 25 ml of stock solution to get a solution of 5 PPM.
6. Repeat above procedure with 30 ml of stock solution to get a solution of 6 PPM.
7. Repeat above procedure with 35 ml of stock solution to get a solution of 7 PPM.
8. Repeat above procedure with 40 ml of stock solution to get a solution of 8 PPM.
9. Repeat above procedure with 45 ml of stock solution to get a solution of 9 PPM.
10. 50 ml of stock solution are diluted with 50 ml deionized water to get a final solution of 10 PPM.

Drawing of Calibration Curves

The Atomic Absorption Spectrophotometer readings are displayed in absorption but they can be readily converted by means of a table to percent absorption which varies almost linearly with concentration. The conversion table is provided in the Analytical Methods Book for Perkins-Elmer Model 403 A.A. spectrophotometer.

The instrument parameters are recorded with each set of data so they can be duplicated when corresponding sample runs are made. Each curve standards is run in ascending order of element concentration. Curves can be conveniently plotted on expanded logarithmic paper

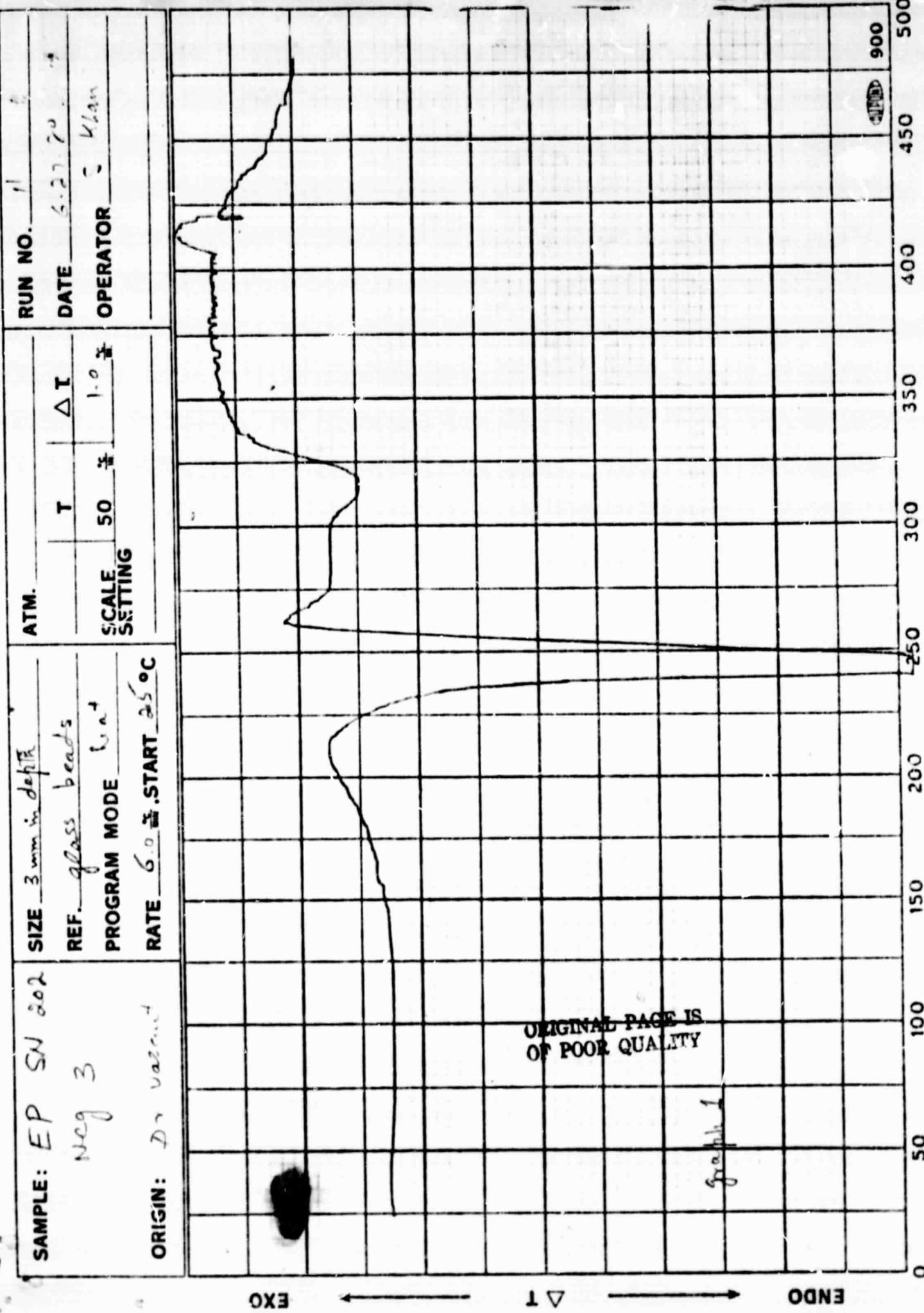
Analysis of Samples

The agreement of the results obtained by A.A Spectroscopic analysis with those obtained by standard analysis have previously been confirmed (Please see annual report 1979).

For analysis of each sample a calibration curve is derived from standard solutions. The given samples are diluted and the concentration of the metal in the aliquot is calculated from the calibration curve. This is multiplied by the dilution factor to give the concentration of the metal in the original sample.

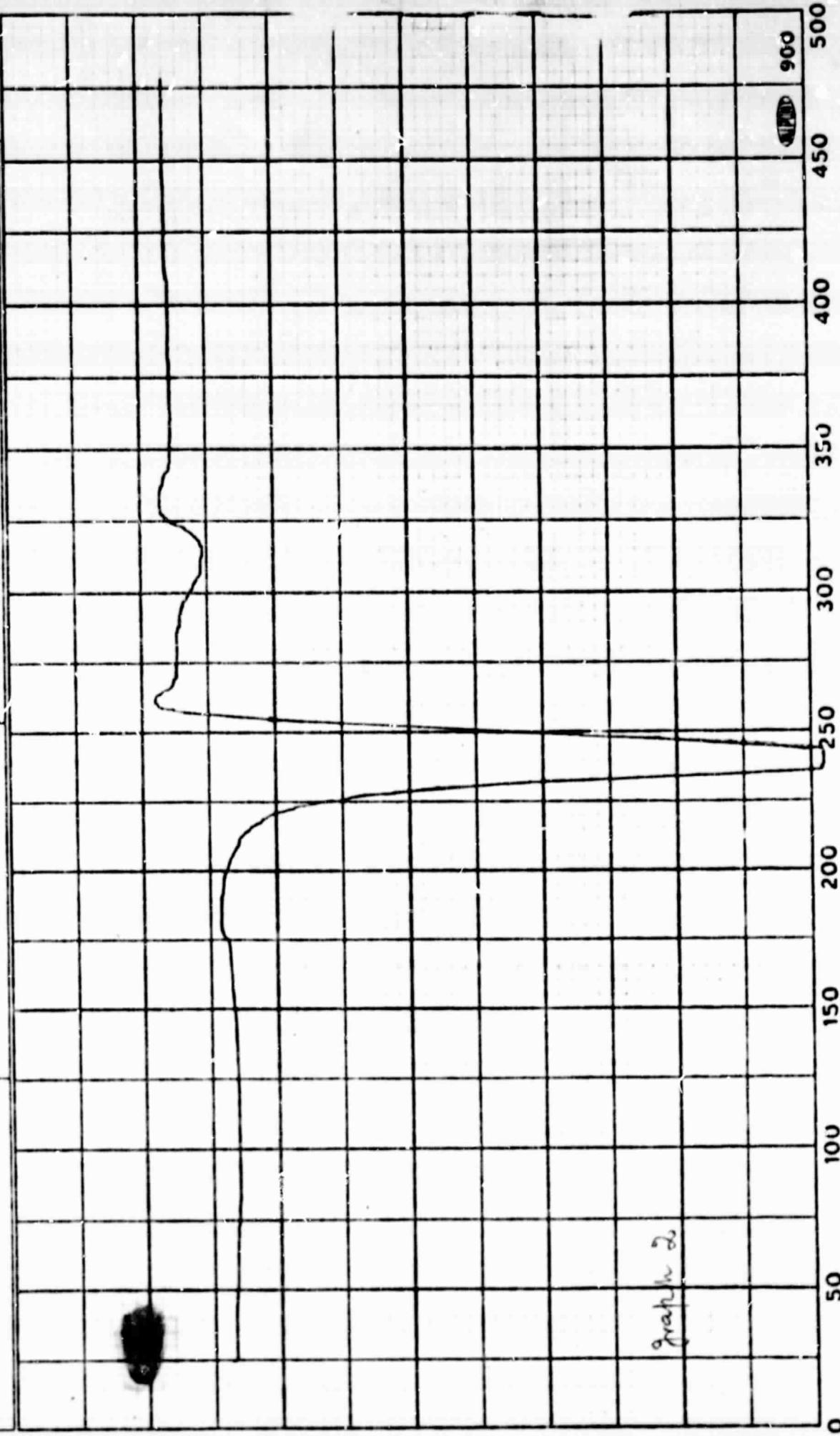
The results obtained are given in table Ia through table XXb. Tables "a" contain the data for standard calibration curve and tables "b" contain the data for analyzed samples.

The points corresponding to each analyzed sample have been marked on the calibration curve.

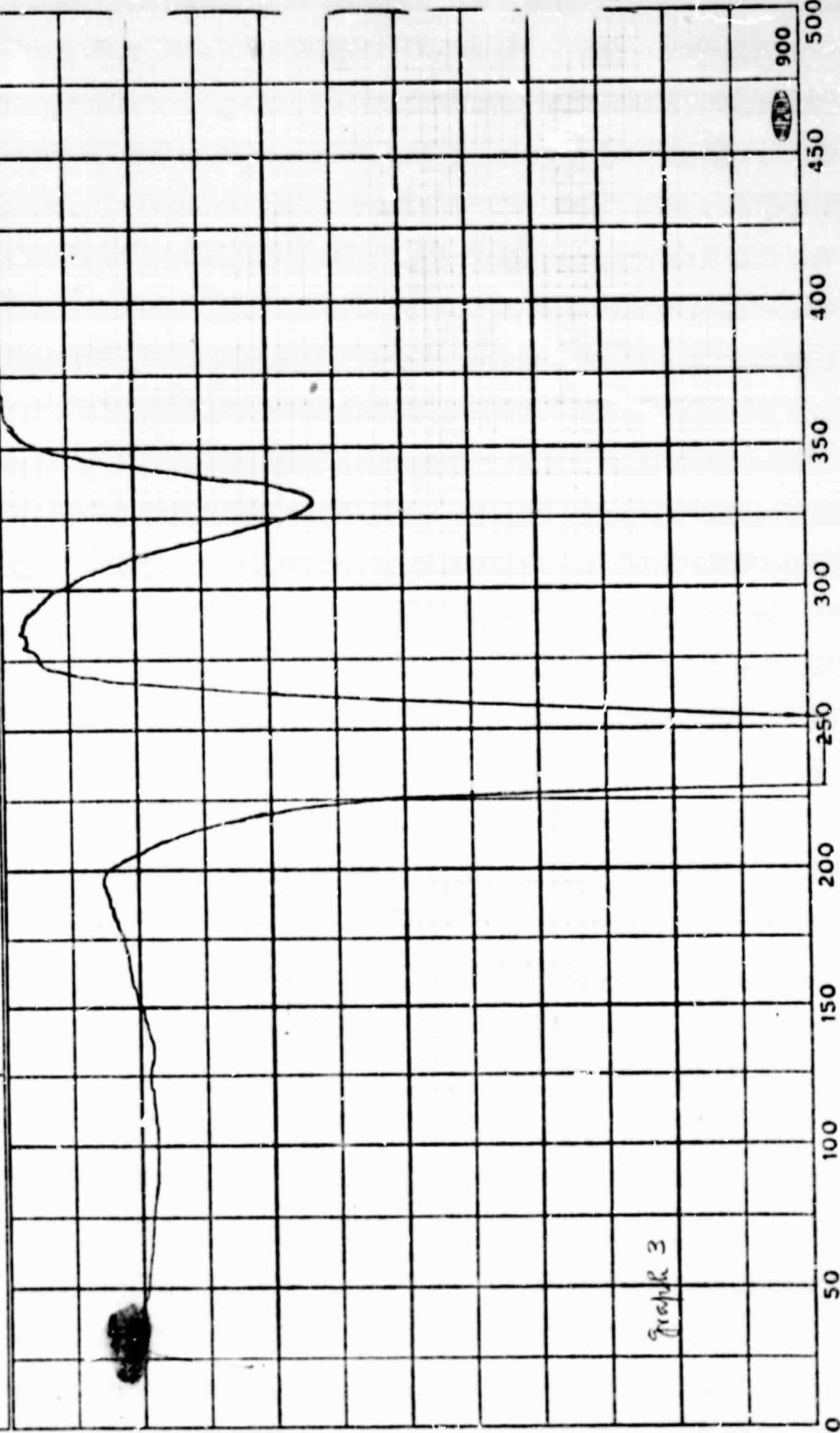


| | | | | | | | |
|--------------|--------------------|---------|----------|------|------------|----------|----------|
| SAMPLE: | Plots No 1 | SIZE | 2 mm dia | ATM. | % | RUN NO. | / |
| REF. | glass bends | | | T | ΔT | DATE | 6.26.00 |
| PROGRAM MODE | Heat | SCALE | 50 | 5 | 1 | OPERATOR | S. K. L. |
| RATE | 7.0 °C/START 25 °C | SETTING | | | | | |

ORIGIN: D Y Vasant

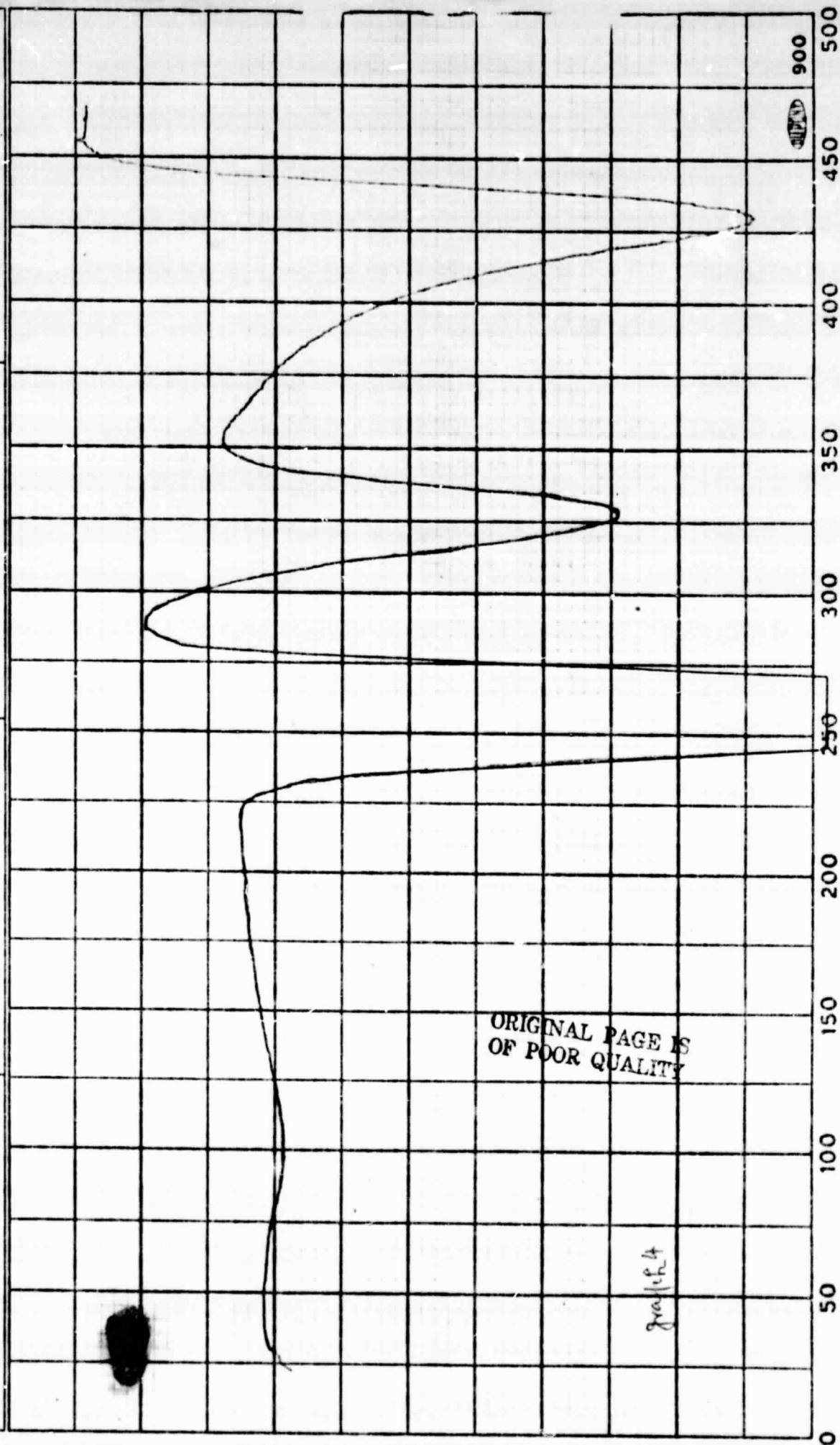


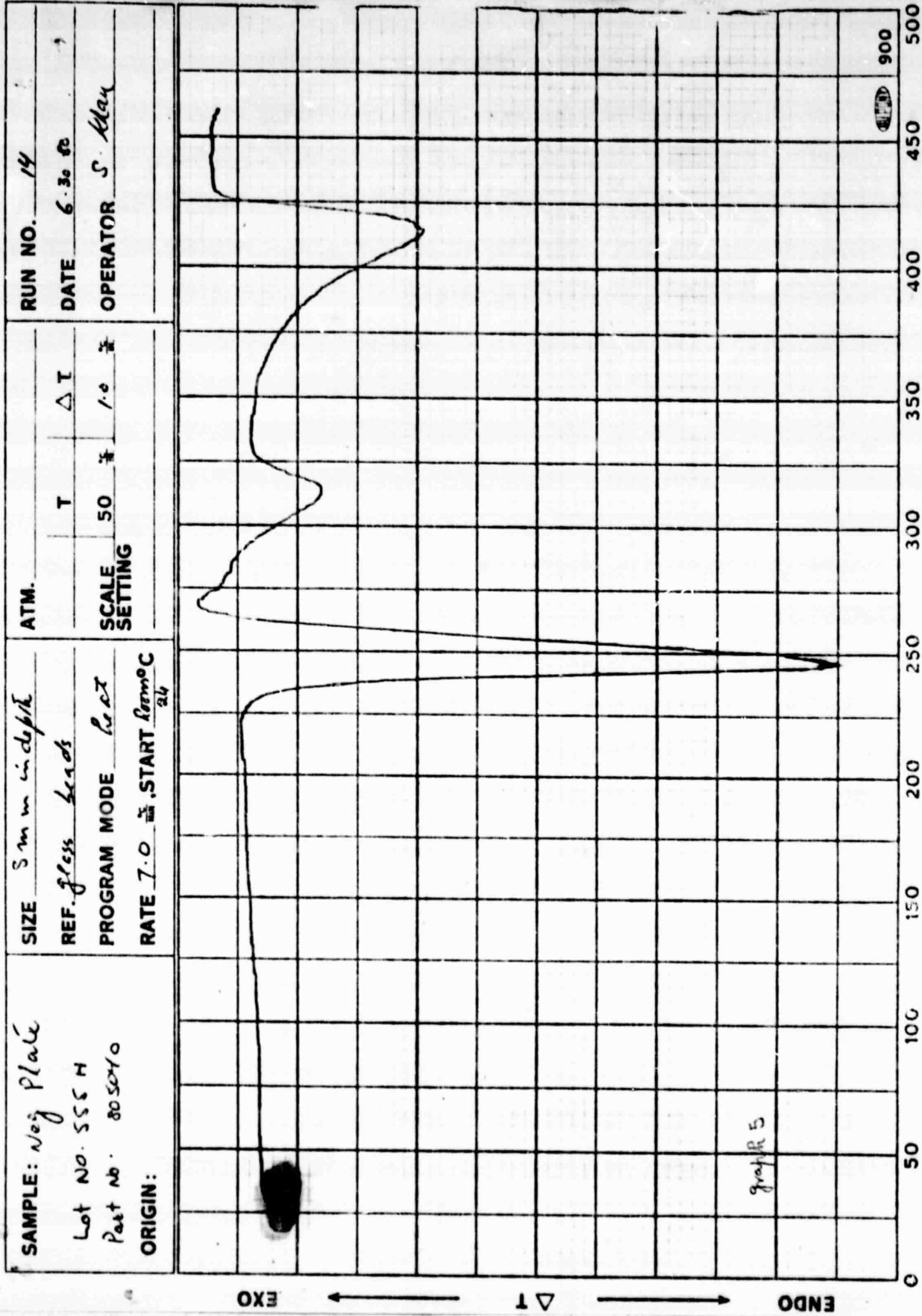
| | | | |
|---|-----------------------------|------------------|-------------------------------------|
| SAMPLE: EP SN 202 | SIZE Δ_{min} in decm | ATM. | RUN NO. #3 |
| REF. glass Beads | | T | DATE 6-24-68 |
| PROGRAM MODE | $\frac{1}{2} \text{ sec}$ | ΔT | OPERATOR S. Klein |
| RATE 15 $\frac{\text{sec}}{\text{deg}}$ | START 25 °C | SCALE SETTING | 1.0 $\frac{\text{sec}}{\text{deg}}$ |
| ORIGIN: $\Delta_t = 0$ | | | |



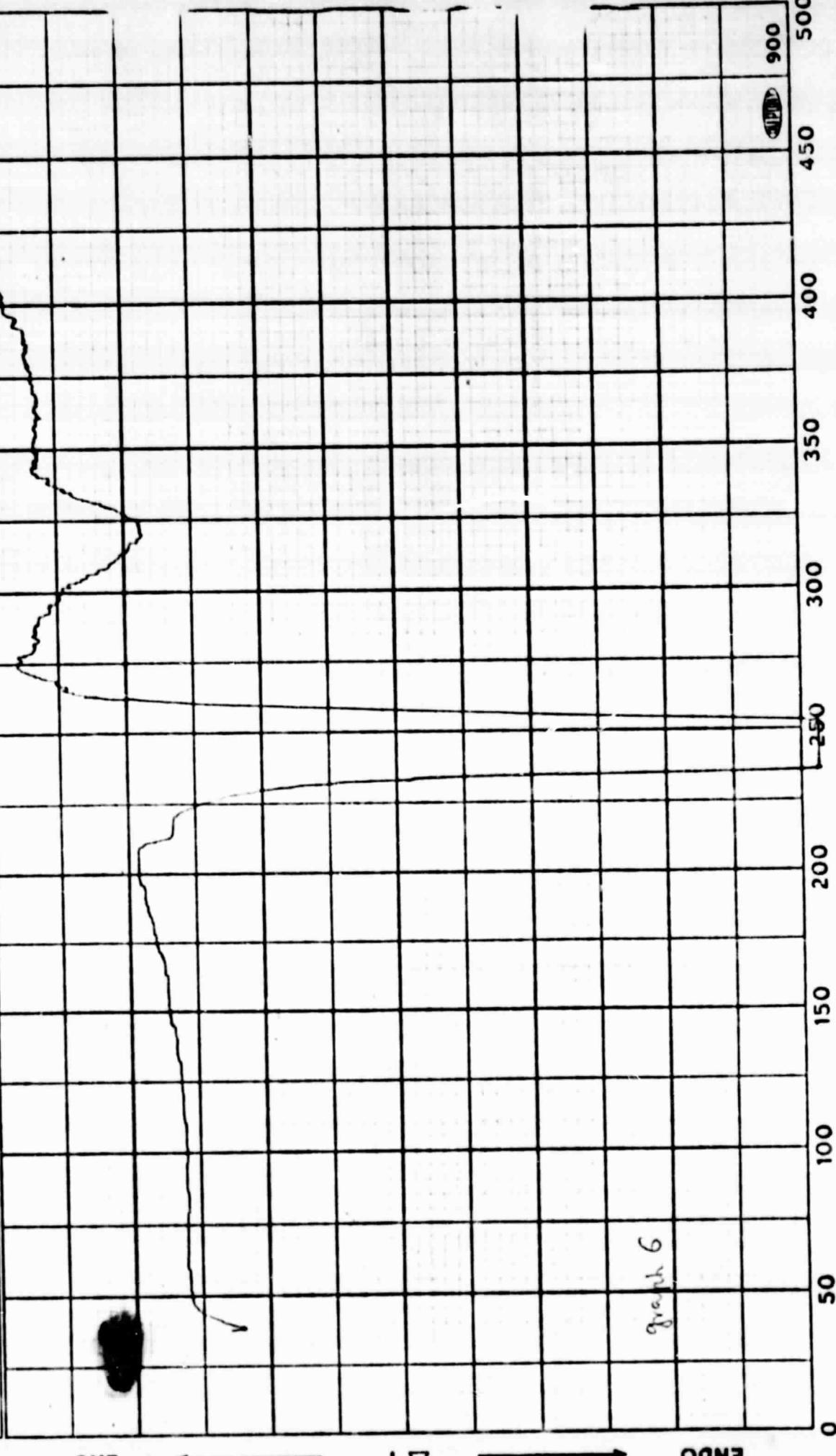
900
450
500

| | | | | | | | |
|---------|------------|--------------|--------------|------------|------------|----------|----------|
| SAMPLE: | 100g Plate | SIZE | 200 mm Depth | ATM. | | RUN NO. | #4 |
| Lot No: | 5554 | REF. | 5557 5558 | T | ΔT | DATE | 6-25-86 |
| Date | 10-20-86 | PROGRAM MODE | Run 4 | SCALE | 50 | OPERATOR | S. K. P. |
| RATE | 5 mm/min | SETTING | 1.0 | ΔT | 1.0 | | |
| ORIGIN: | START | 25 °C | | | | | |

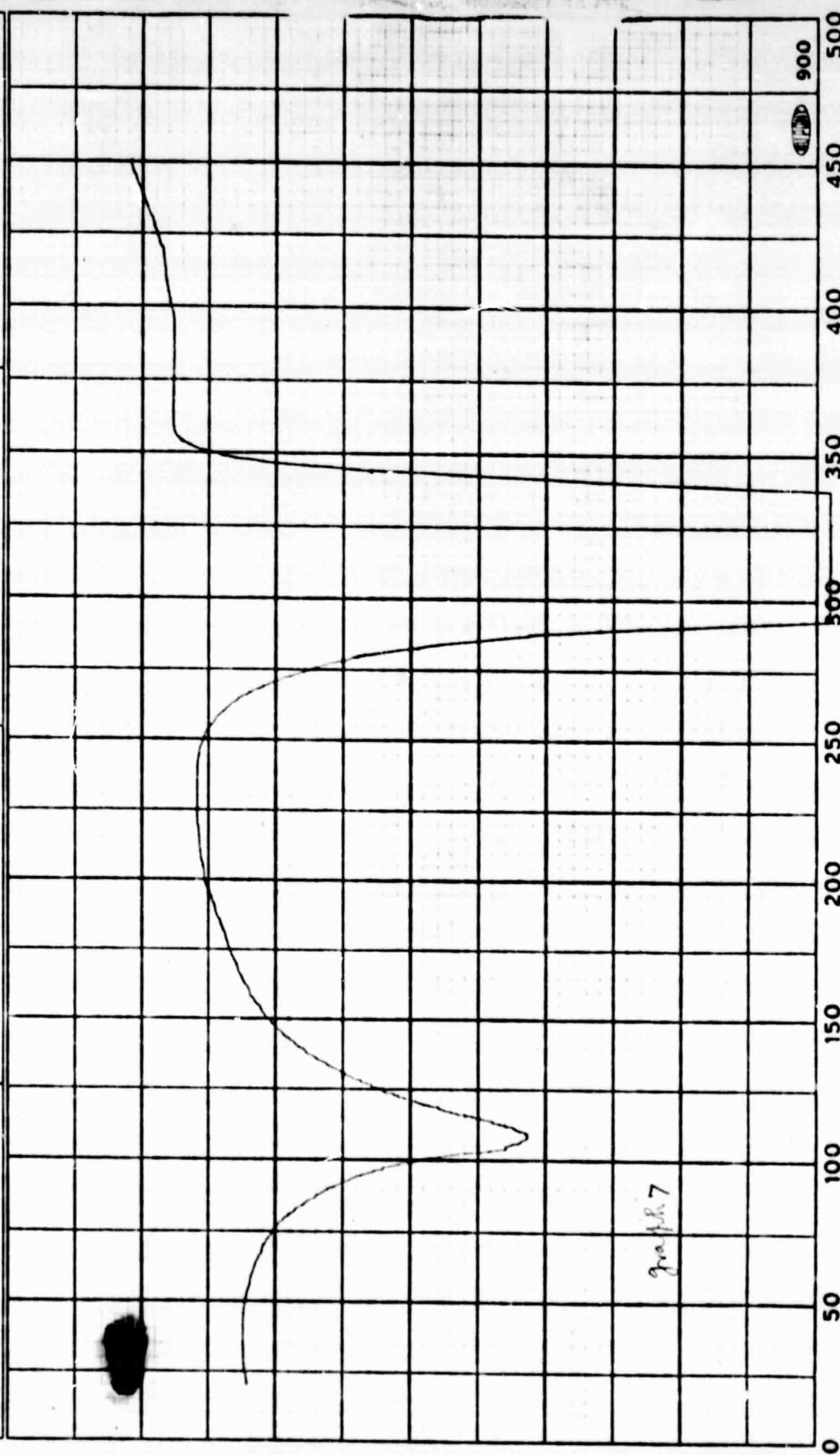


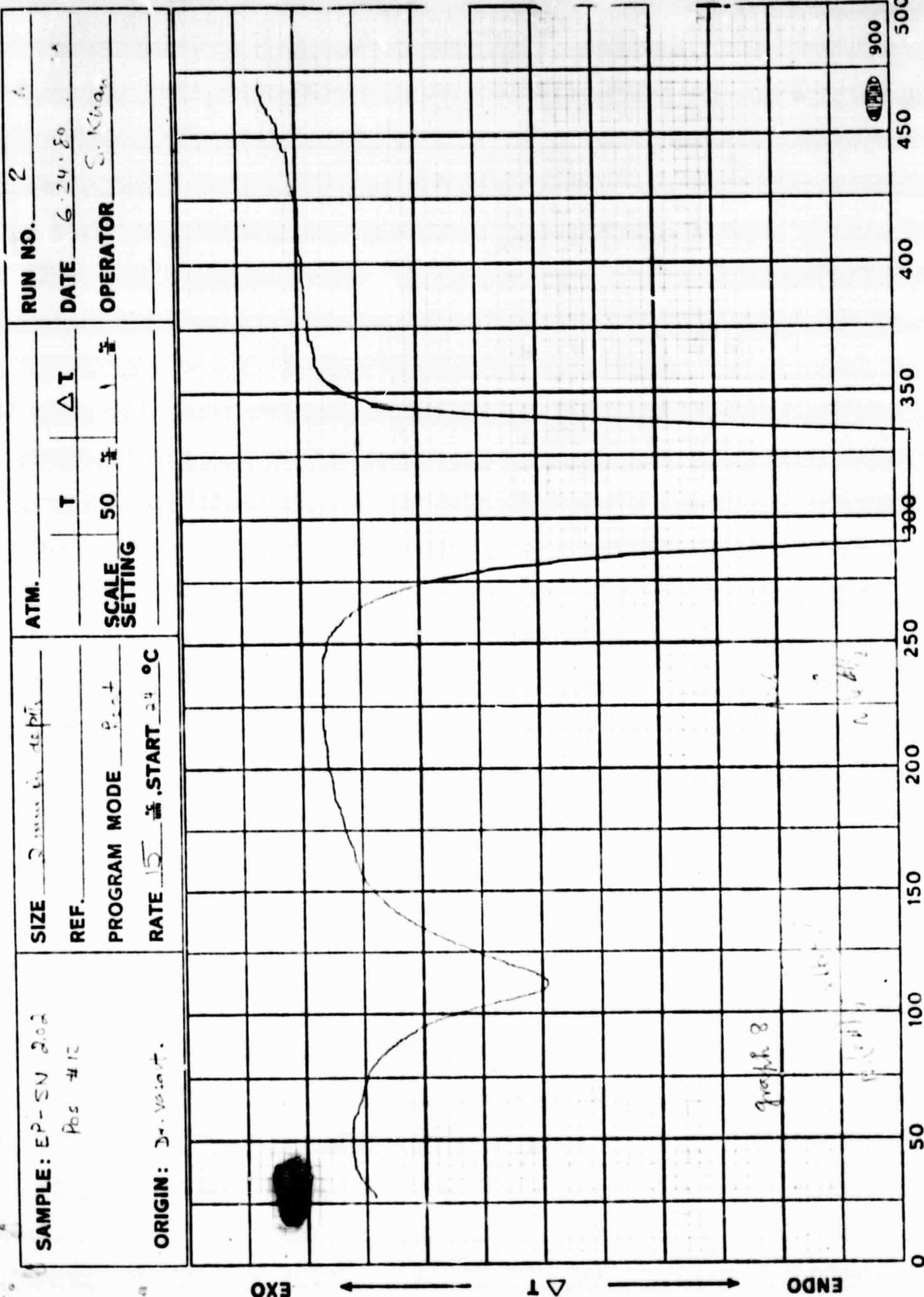


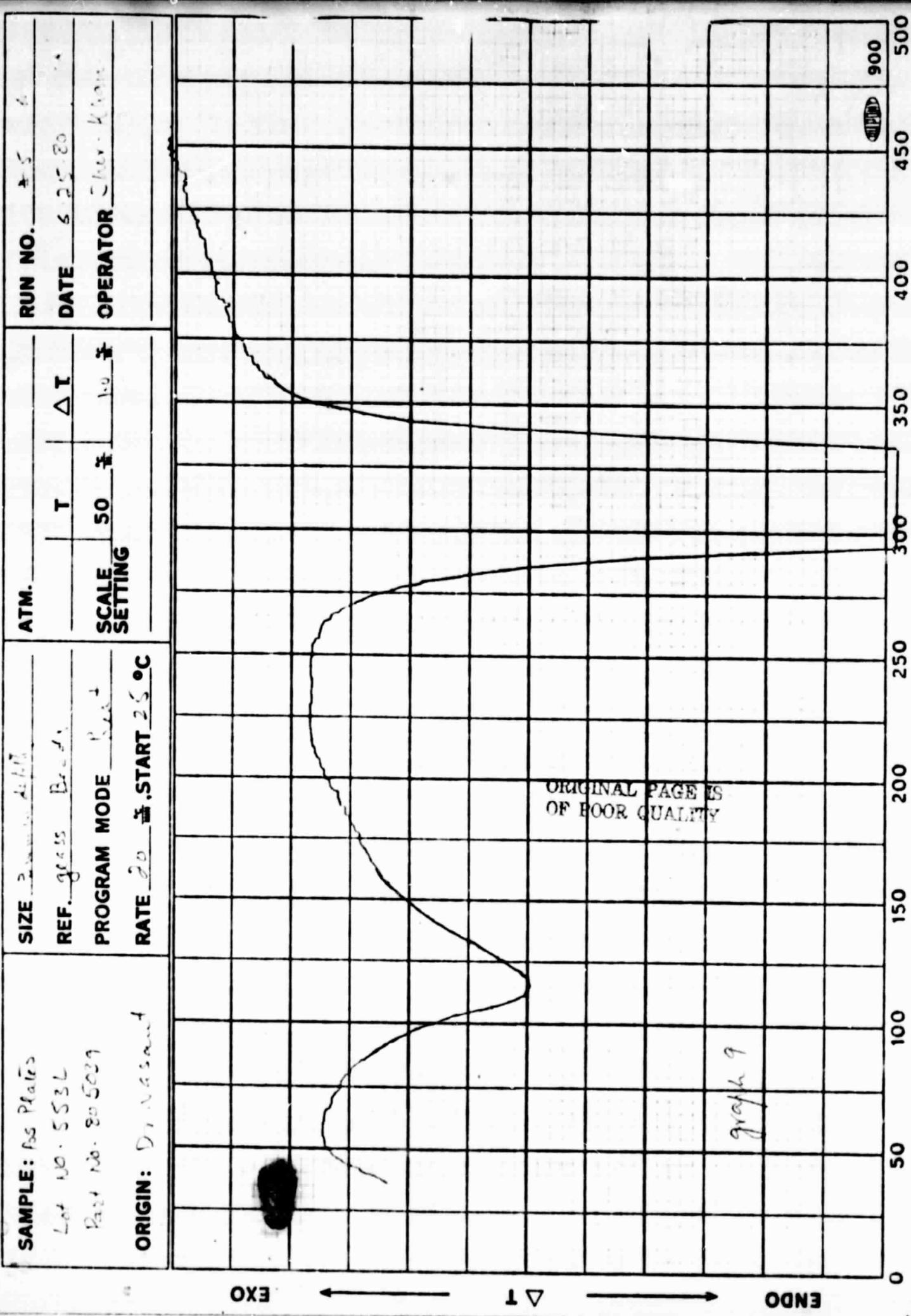
| | | | |
|--------------|-------------|----------|---------|
| SAMPLE: | 45001 | RUN NO. | # 6 |
| SIZE | 10.00 ± 1.7 | DATE | 6.25.80 |
| REF. | 10.00 ± 1.7 | OPERATOR | John |
| PROGRAM MODE | Load | | |
| RATE | 10 °/min | START | 25°C |
| ORIGIN: | | | |



| | | | |
|--------------------------------|----------------------------|---------------------|------------------------|
| SAMPLE: EP - SN 202 Pcs # 2 | SIZE <u>2 mm disc</u> | ATM. | RUN NO. <u>1</u> |
| REF. <u>Alumina</u> | | | DATE <u>6.24.69</u> |
| PROGRAM MODE <u>P2A4</u> | SCALE <u>50</u> | ΔT <u>1</u> | OPERATOR <u>S. Kue</u> |
| RATE <u>15</u> | SETTING <u>START 24 °C</u> | | |
| ORIGIN: D. U. L. A. | | | |



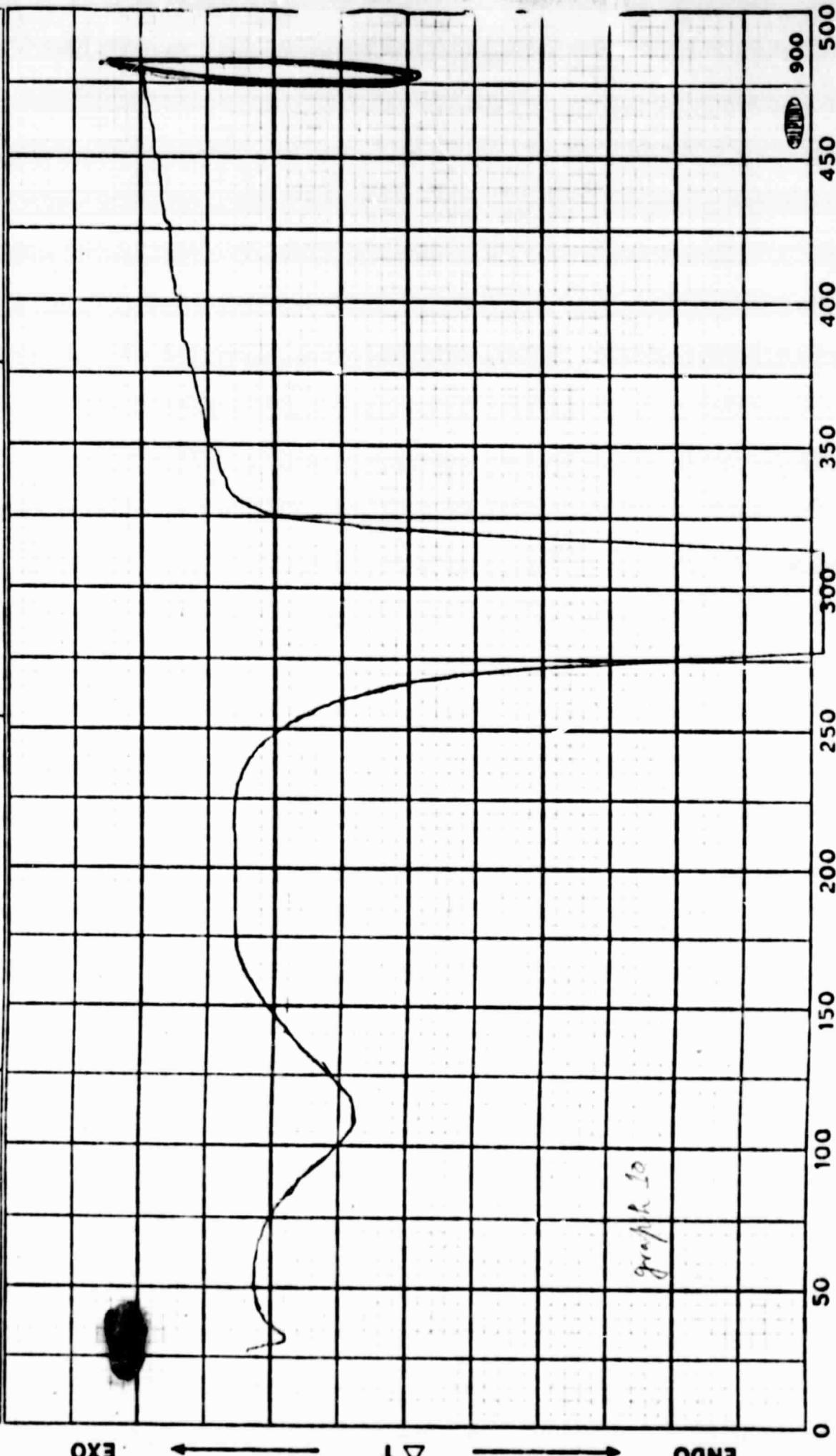




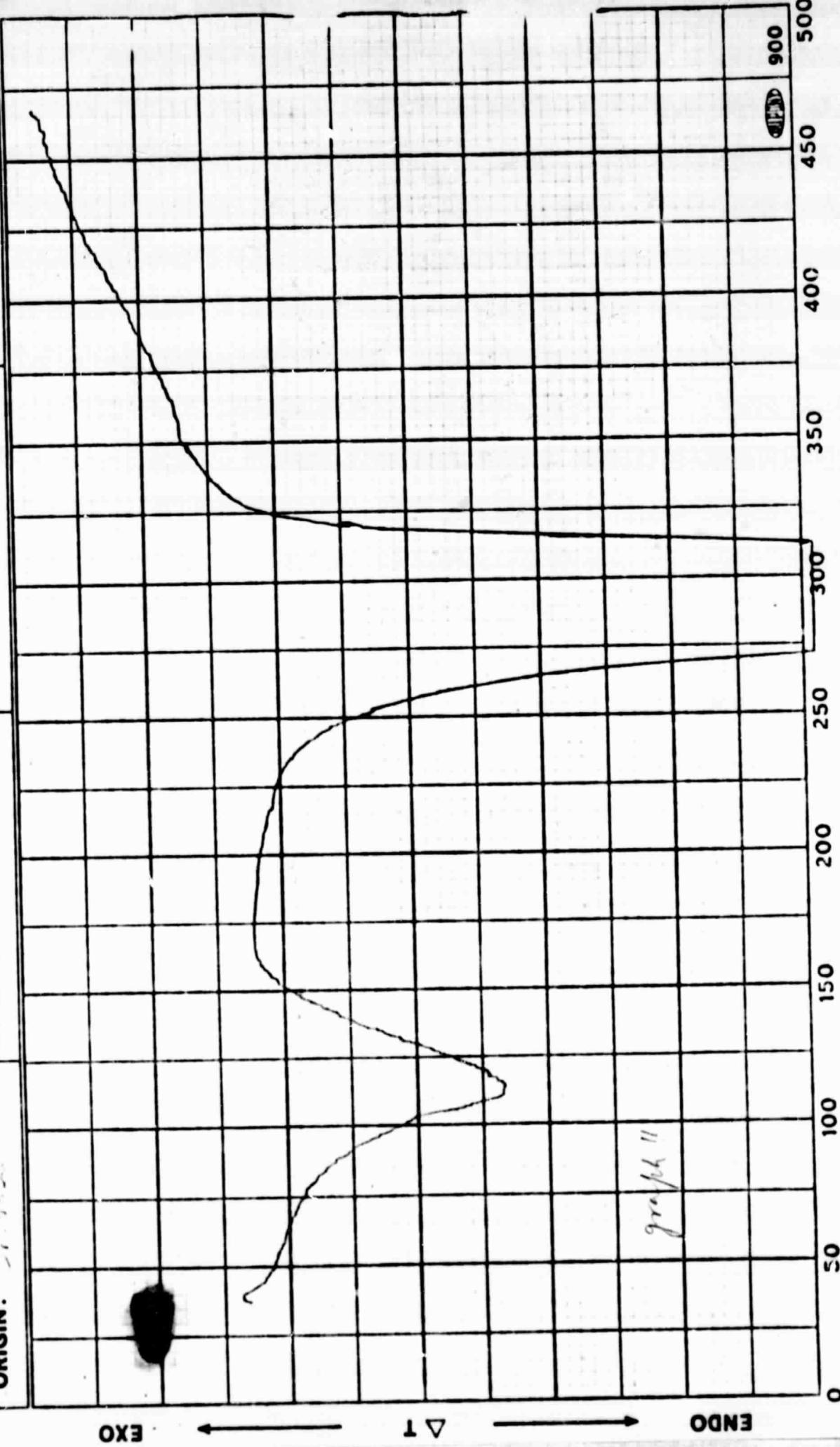
| | | | |
|-----------------------------|-----------------------|-------------------|------------------------|
| SAMPLE: <i>PoC</i> | SIZE <i>2 mm dia.</i> | ATM. | RUN NO. <i>#7</i> |
| REF. <i>Glass beads</i> | | T | DATE <i>6.26.85</i> |
| PROGRAM MODE <i>Program</i> | | ΔT | OPERATOR <i>C.K.C.</i> |
| RATE <i>15 °C/min</i> | SCALE <i>50</i> | SETTING <i>10</i> | |

Lan *100* *200* *300* *400* *500*

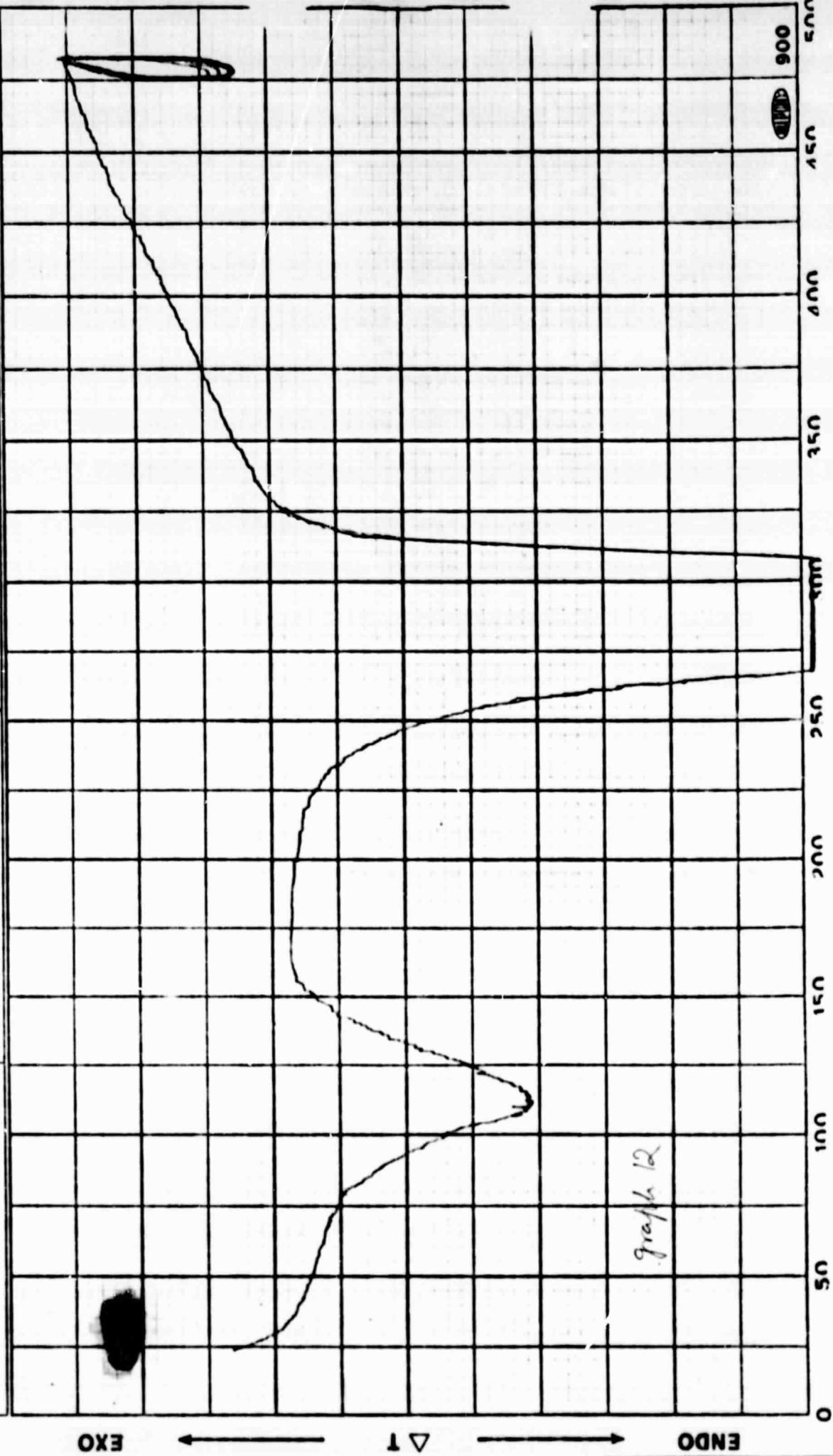
ORIGIN:

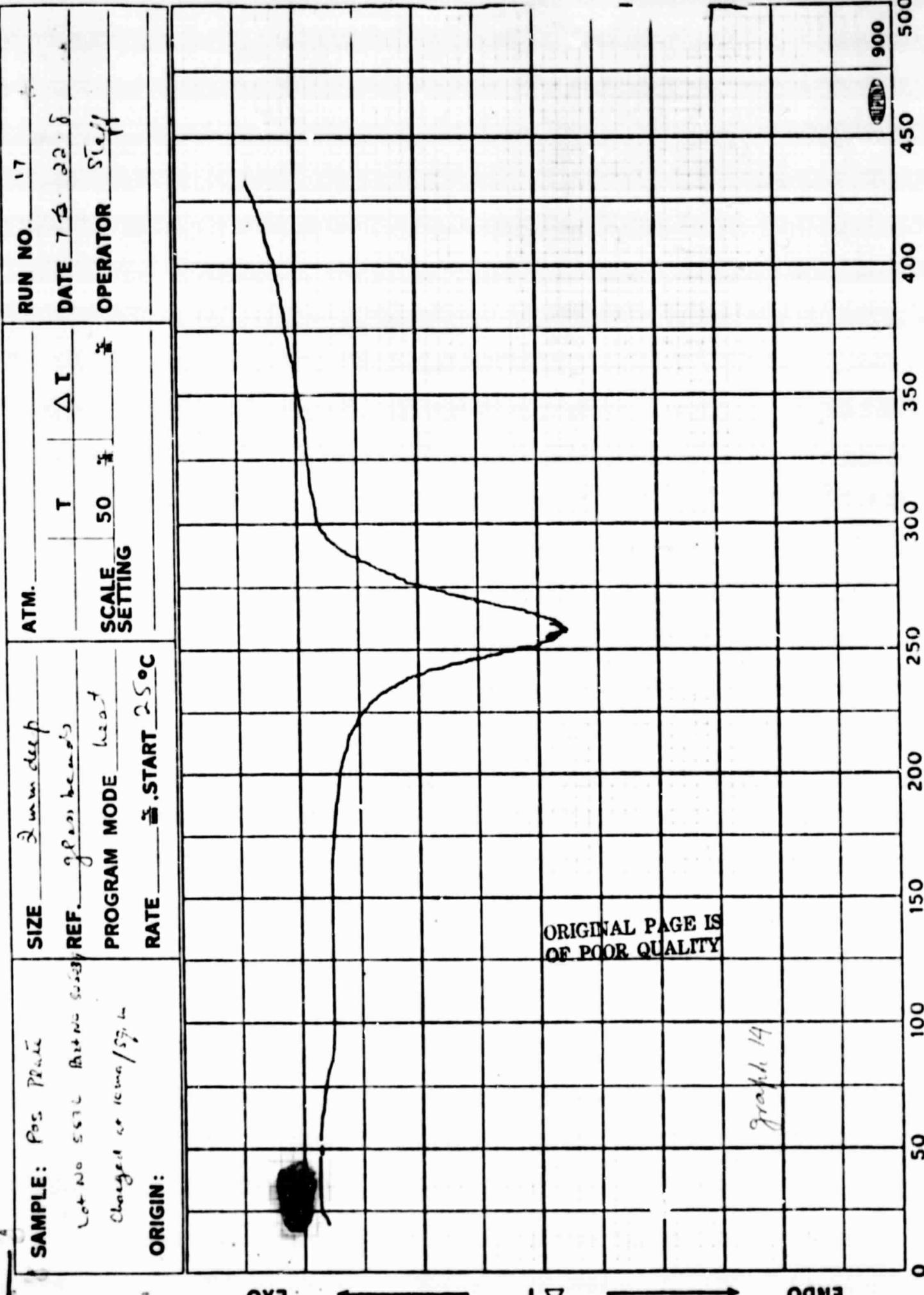


| | | | |
|--------------------|----------------|-------------------|-----------|
| SAMPLE: TDR 5 | SIZE 2 mm dia. | ATM. | RUN NO. 2 |
| REF. Alumina Beads | | DATE 6/25/80 | |
| PROGRAM MODE Heat | SCALE 50 | OPERATOR S. K. M. | |
| RATE 15 °/min | SETTING 1.0 | | |
| ORIGIN: 200 | | | |

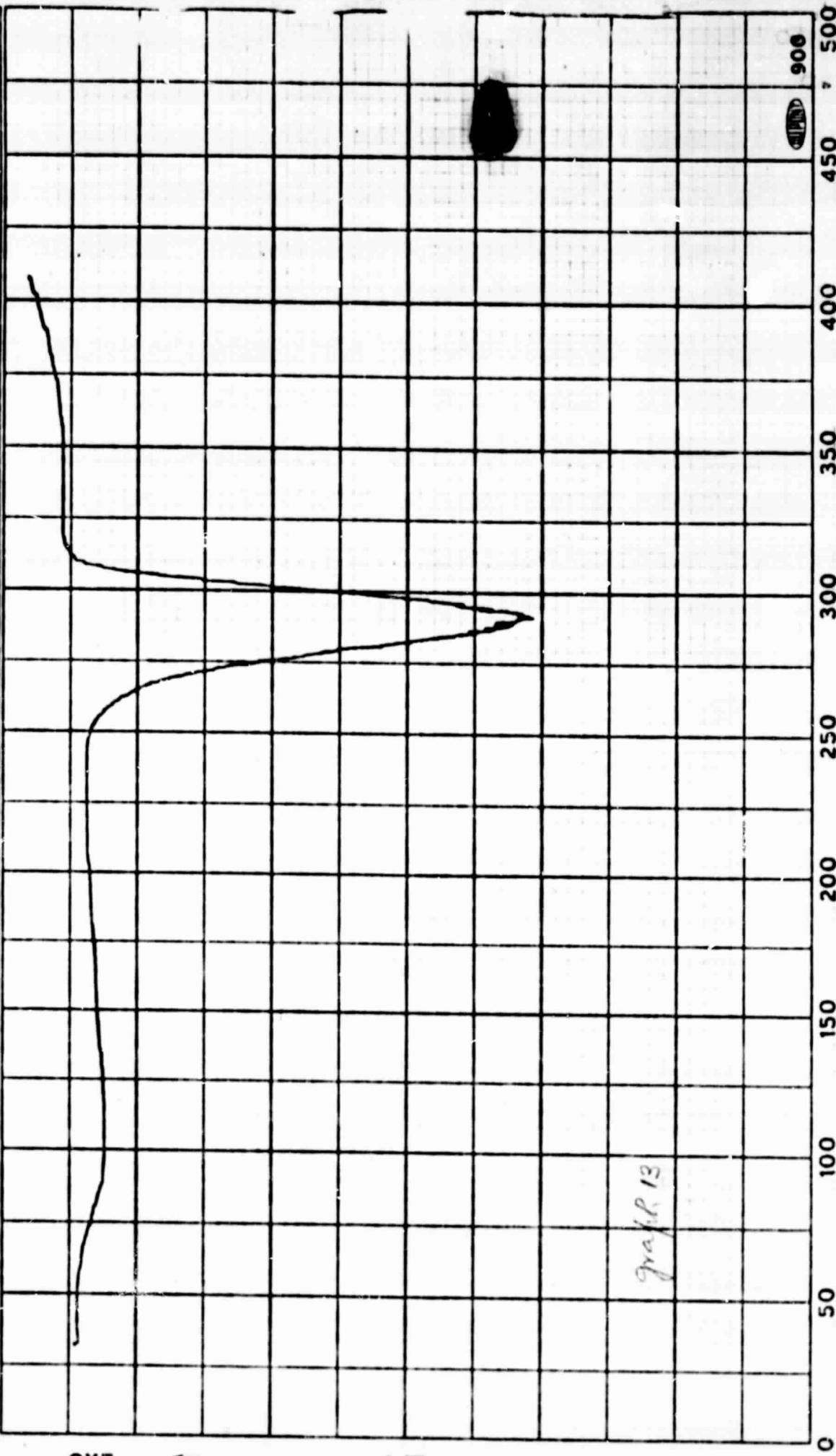


| | | | |
|--|-----------------------|------------|-------------------------|
| SAMPLE: TDRS 2 | SIZE <u>2 mm dia.</u> | ATM. | RUN NO. <u>9</u> |
| REF. <u>3145: blend</u> | | T | DATE <u>6-26-80</u> |
| PROGRAM MODE <u>peak</u> | SCALE <u>50</u> | ΔT | OPERATOR <u>J. Chau</u> |
| RATE <u>15</u> $\frac{\text{m}}{\text{sec}}$ | SETTING <u>5 sec</u> | | |
| ORIGIN: <u>downward</u> | | | |

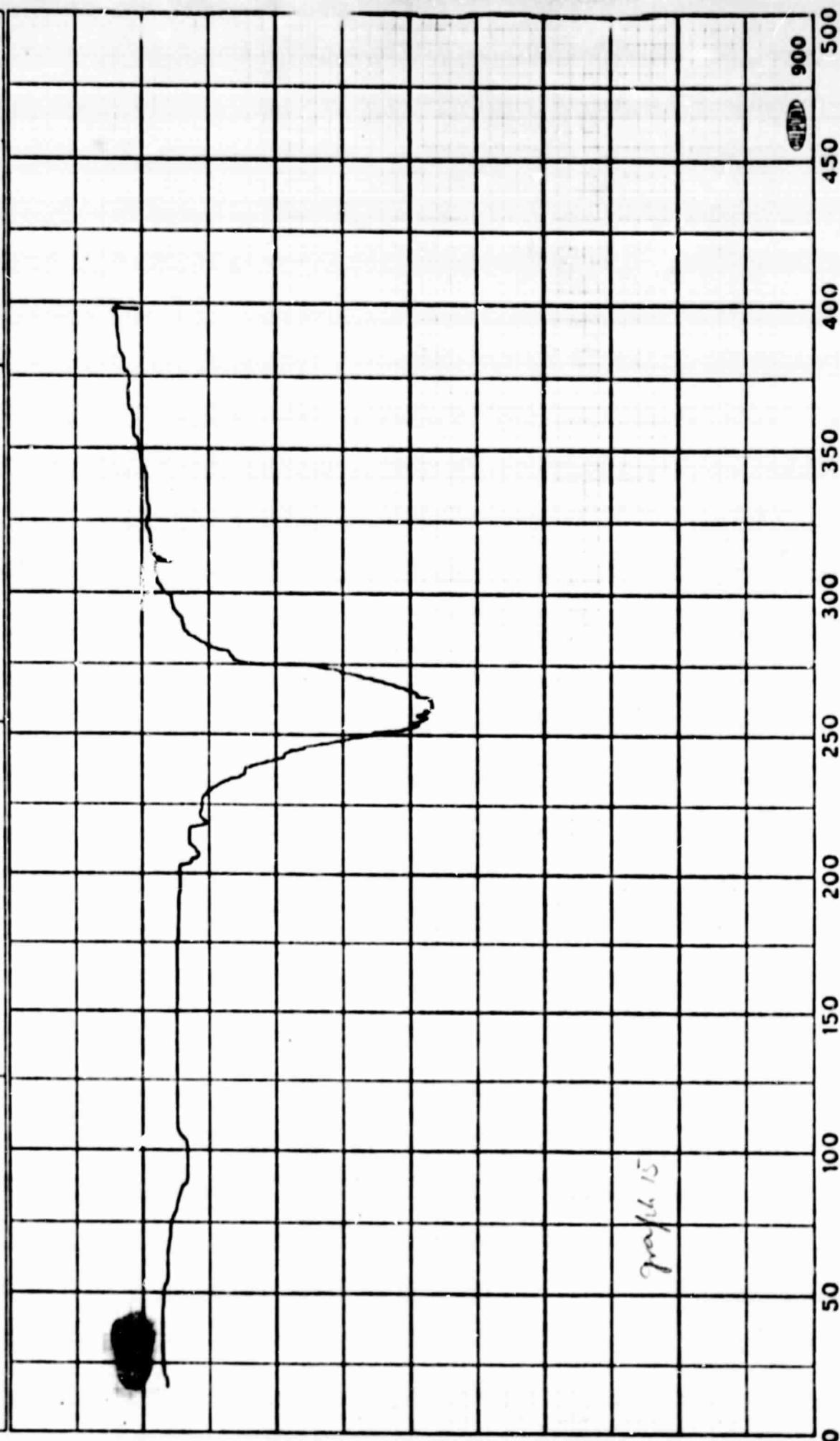


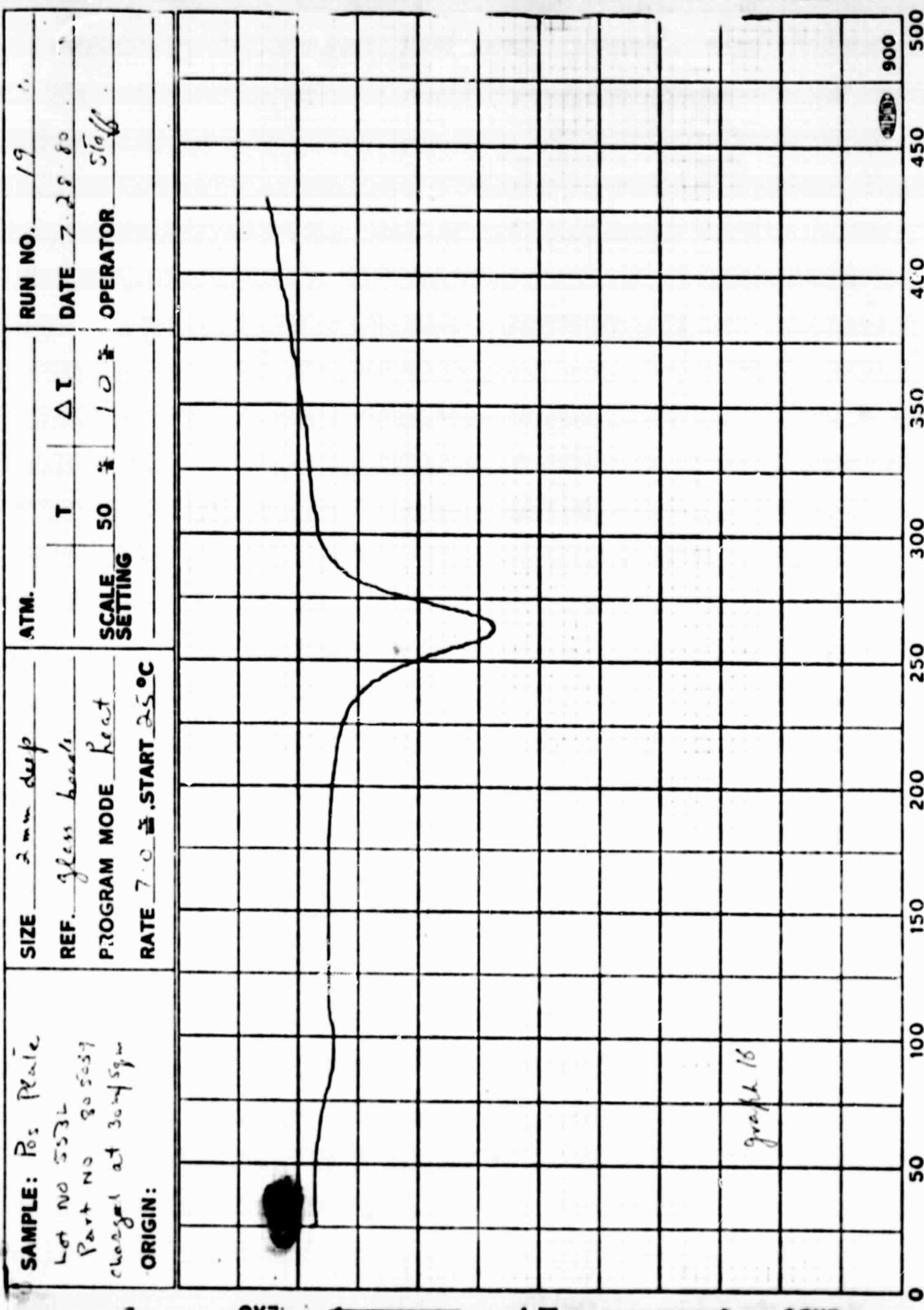


| | | | |
|--------------------------|---------------------------|------------|-----------------------|
| SAMPLE: <i>Pcs. Plas</i> | SIZE <i>dimin. length</i> | ATM. | RUN NO. #16 |
| <i>Loe NC 5531</i> | <i>REF. Glass beads</i> | T | DATE 7-21-80 |
| <i>Pcs. NC 80563-1</i> | PROGRAM MODE <i>heat</i> | ΔT | OPERATOR <i>Staff</i> |
| ORIGIN: | SCALE SETTING | 50 | |
| RATE <i>25 °C/min</i> | .START <i>25 °C</i> | 10 | |

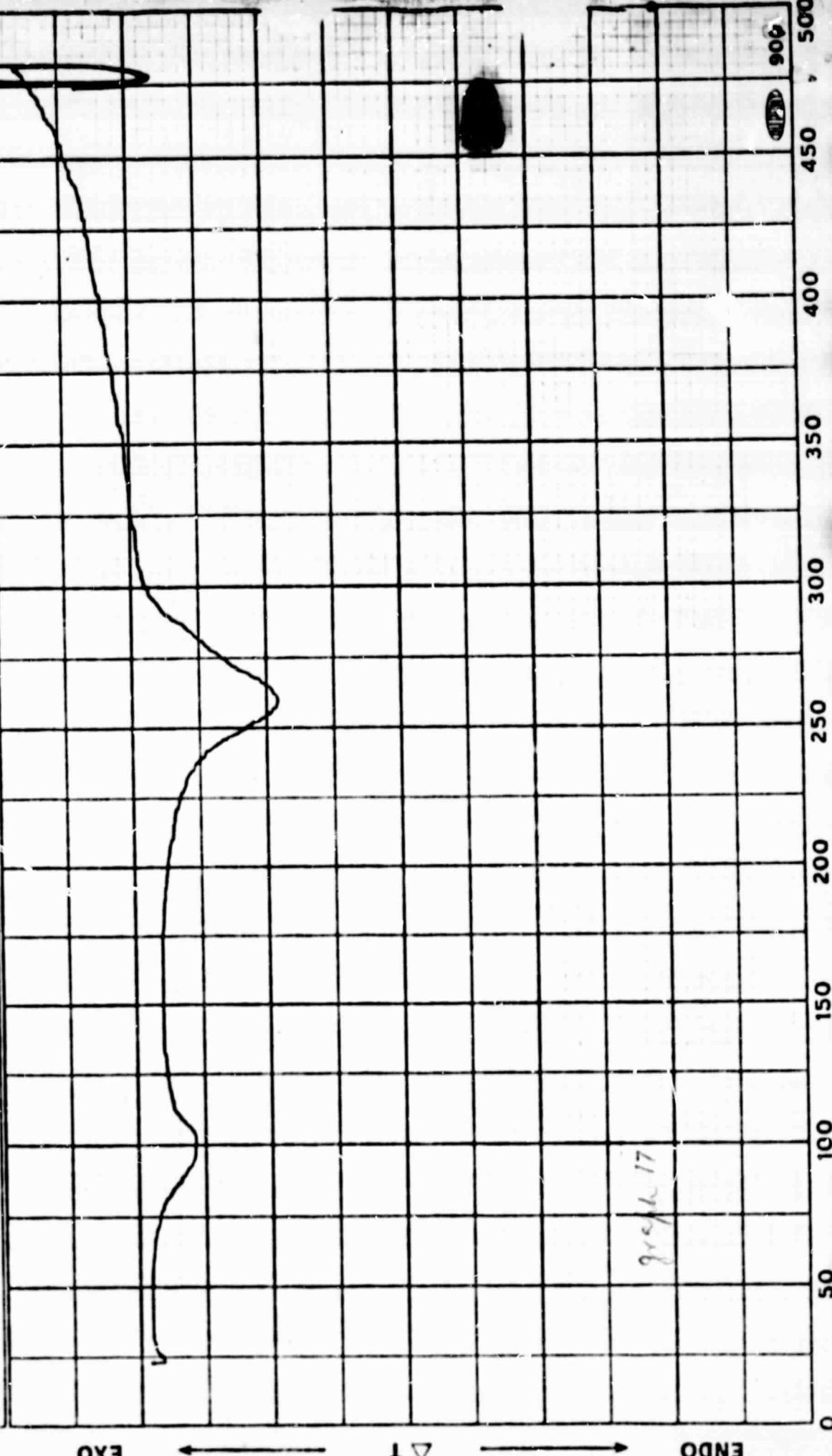


| | | | |
|---------------------------|----------------------|------------|-----------------------|
| SAMPLE: Pcs Blue | SIZE 3 mm disc | ATM. | RUN NO. 14 |
| Lot No 5534 | REF. glass beads | T | DATE 7.24.80 |
| Part No 86557 | PROGRAM MODE heat | ΔT | OPERATOR <u>Steve</u> |
| Cleaned at 30 min / 50 °C | RATE <u>5</u> °C/min | SCALE 50 | |
| ORIGIN: | SETTING 10 | 10 | |





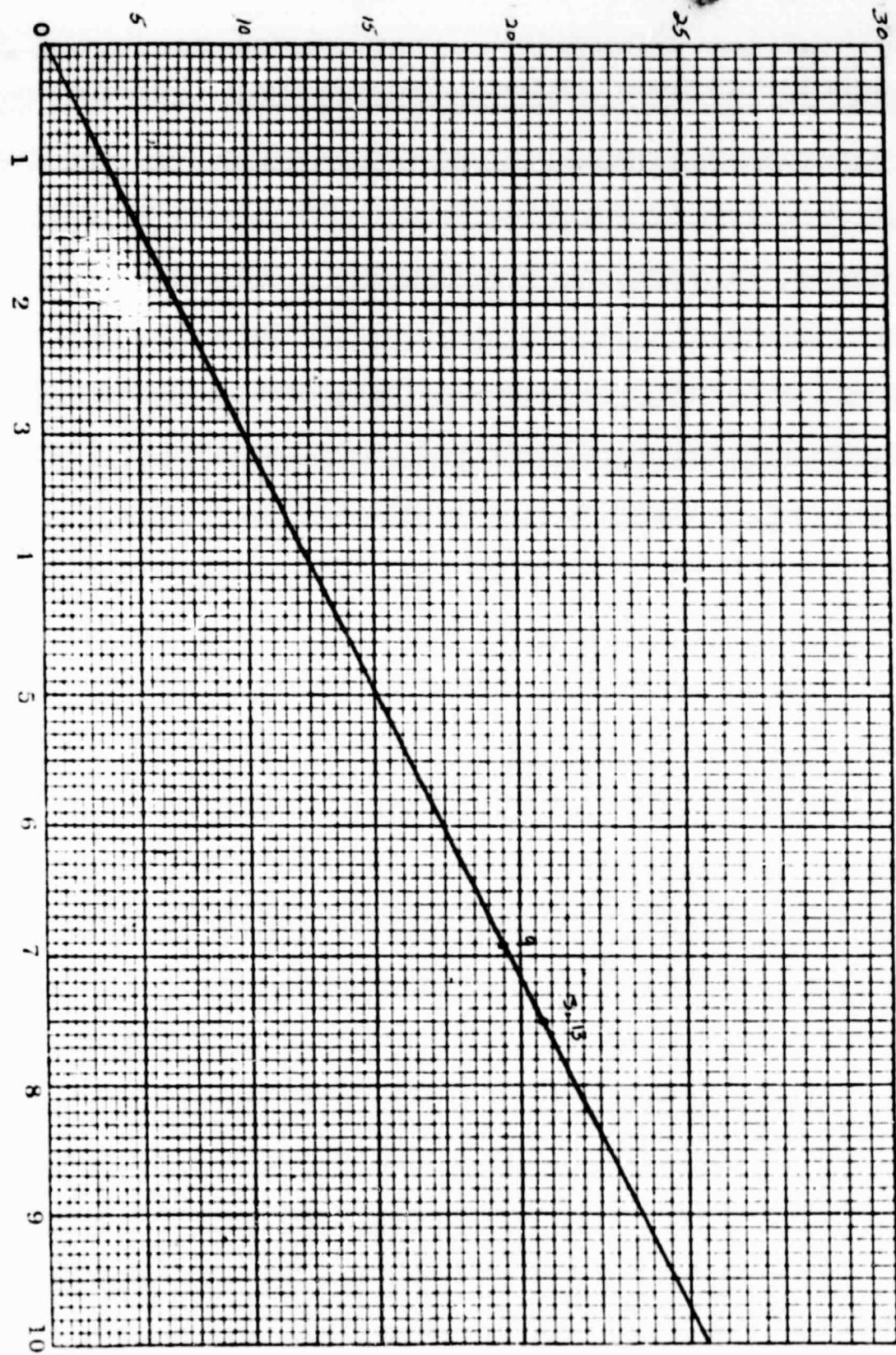
| SAMPLE: Pos Plate | SIZE | ATM. | RUN NO. |
|--------------------------------------|-----------------|------------|----------|
| REF. | T | Δ T | DATE |
| PROGRAM MODE | SCALE | SO | OPERATOR |
| Part No 553L Charged at 40 m/s/in | 10 | 10 | Stall |
| ORIGIN: | RATE 7.0 in/SEC | START 2.0C | |



ABSORPTION, percent on A.A. Spectrophotometer

Analysis of samples G.E. 12 and CN 22 #3, #7, and #13.

graph 1



Data for Graph I

Analysis of cell GE 12AM S/N01 plates #3, #9, #13

Calibration curve for Ni.

Table Ia

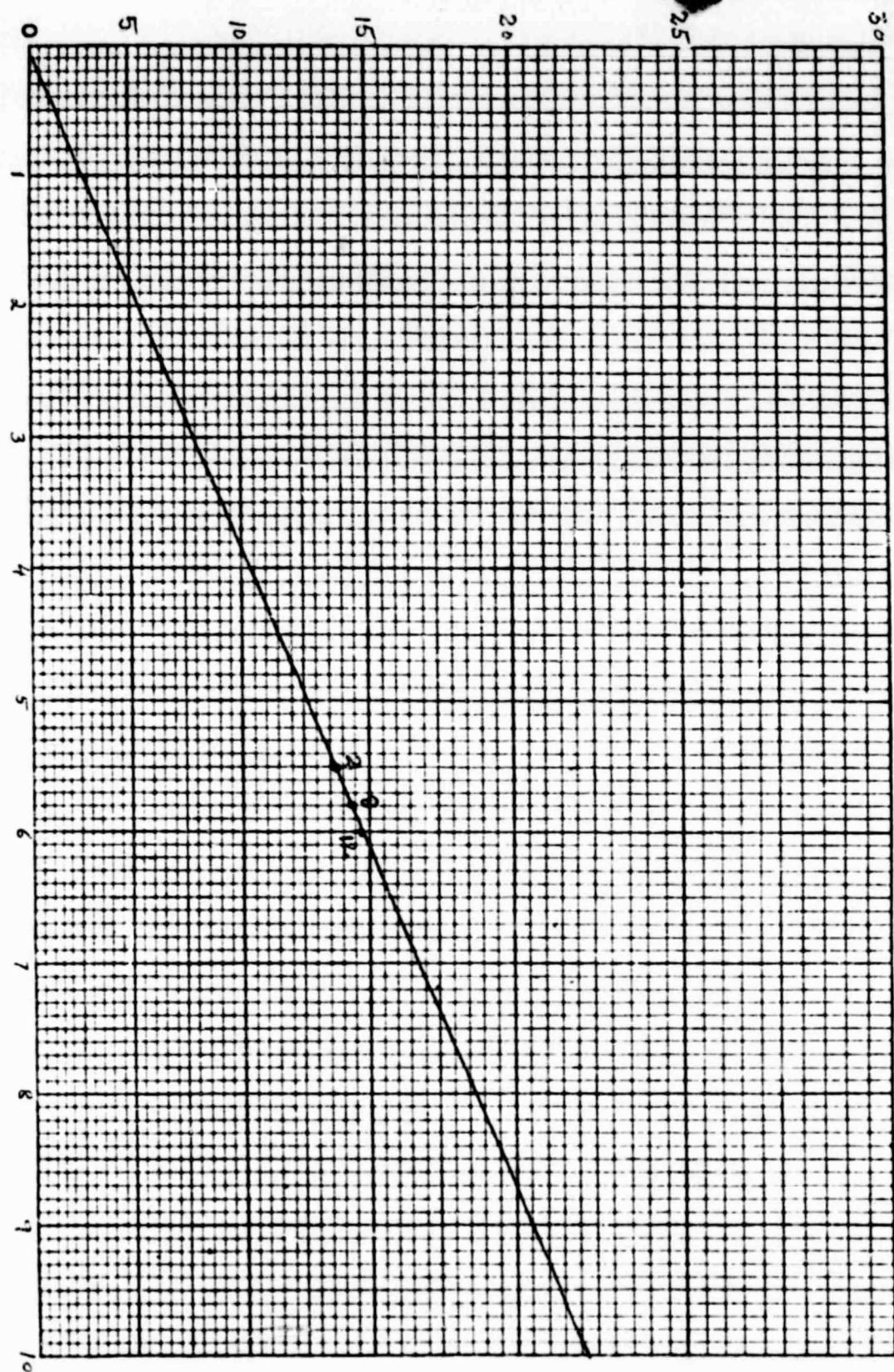
| PPM | A.A. Reading | %Abs |
|-----|--------------|------|
| 2 | .0234 | 5.3 |
| 4 | .044 | 9.7 |
| 6 | .0644 | 13.8 |
| 8 | .0842 | 17.6 |
| 10 | .1011 | 22.4 |

Unknown sample analysis

Table Ib

| Sample No. | Dilution factor | A.A. reading | %Abs | PPM | PPM Orig solution |
|-----------------------|-----------------|--------------|------|-----|-------------------|
| GE 12AM S/N01 #3 X 10 | | .079 | 16.7 | 7.5 | 75.0 |
| GE 12AM S/N01 #9 X 10 | | .0734 | 15.6 | 6.9 | 69.0 |
| GE 12AM S/N01#13 X 10 | | .0802 | 16.9 | 7.5 | 70.0 |

ABSORPTION, percent



Analysis of samples GE #2 Positive #2, #8, and #12.

Graph 2

Data for Graph II

Ni analysis of cell GE 02 plates #2, #8, #12

Calibration curve for Ni

Table IIa

| PPM | A.A. Reading | %Abs |
|-----|--------------|------|
| 2 | .030 | 8.0 |
| 4 | .056 | 12.2 |
| 6 | .082 | 17.3 |
| 8 | .109 | 22.2 |
| 10 | .128 | 25.5 |

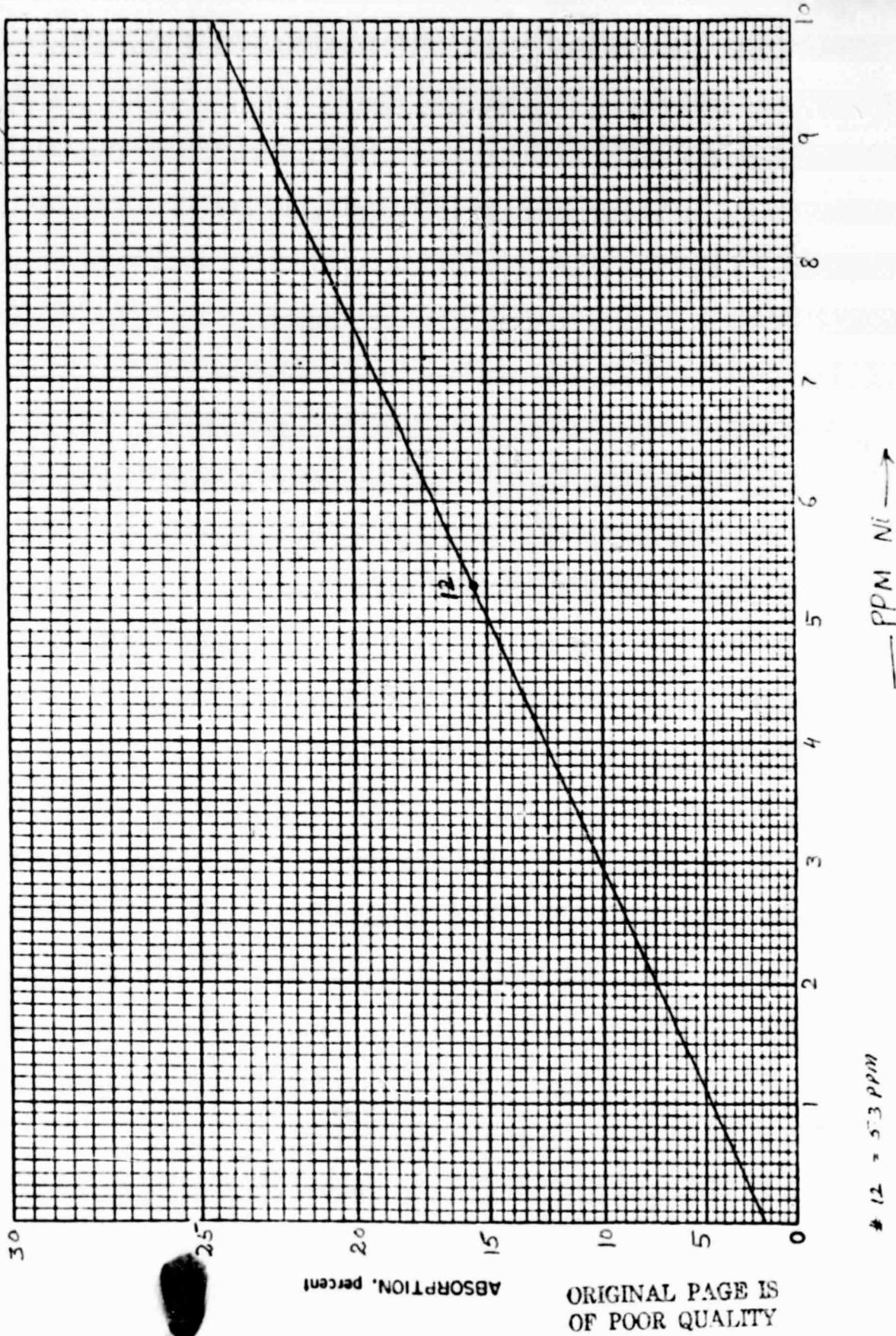
Unknown sample analysis

Table IIb

| Sample No. | Dilution factor | A.A. reading | %Abs | PPM | PPM Orig solution |
|------------|-----------------|--------------|------|------|-------------------|
| GE 02 #2 | 250 | .076 | 16.1 | 5.51 | 1380 |
| " #8 | " | .081 | 17.0 | 5.80 | 1480 |
| " #12 | " | .084 | 17.6 | 6.00 | 1500 |

Analysis of Sample GE 02 Reference #12

Graph 3



Data for Graph III

Ni analysis of cell GE 02 Positive plate #12

Caliberation curve for Ni

Table IIIa

| PPM | A.A. Reading | %Abs |
|-----|--------------|------|
| 2 | .0326 | 7.2 |
| 4 | .576 | 12.4 |
| 6 | .0834 | 17.5 |
| 8 | .1088 | 22.3 |
| 10 | .130 | 25.9 |

Unknown sample analysis

Table IIIb

| Sample No. | Dilution factor | A.A reading | %Abs | PPM | PPM Orig solution |
|----------------|-----------------|-------------|------|------|-------------------|
| GE 02 Pos. #12 | .250 | .0745 | 15.8 | 5.30 | 1320. |

Data for Graph IV

Ni analysis of cell GE 02 plates #3, #9, #13

Caliberation curve for Ni

Table IVa

| PPM | A.A. Reading | %Abs |
|-----|--------------|------|
| 2 | .021 | 4.7 |
| 4 | .0398 | 7.8 |
| 6 | .0622 | 13.3 |
| 8 | .0802 | 15.9 |
| 10 | .0920 | 19.2 |

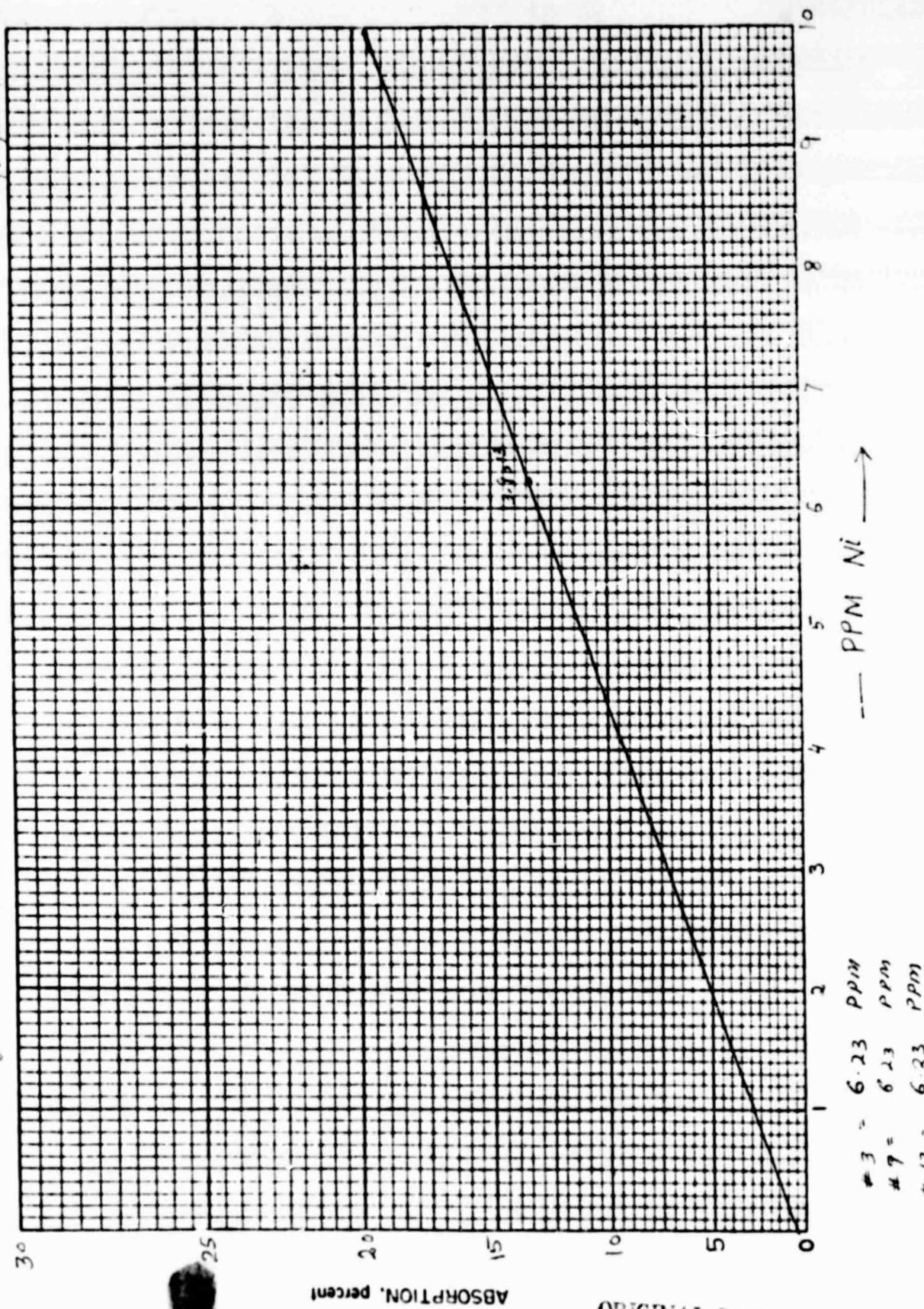
Unknown sample analysis

Table IVb

| Sample No. | Dilution factor | A.A. reading | %Abs | PPM | PPM Orig solution |
|-------------|-----------------|--------------|------|------|-------------------|
| GEO AMP #3 | 250 | .060 | 12.9 | 6.23 | 1557.0 |
| GEO AMP #9 | 250 | .060 | 12.9 | 6.23 | 1557.0 |
| GEO AMP #13 | 250 | .060 | 12.9 | 6.23 | 1557.0 |

Analysis of samples GE 02 AND 03, #9, #13.

Graph 4



ABSORPTION, percent

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Data for Graph IV

Ni analysis of cell GE 02 plates #3, #9, #13

Caliberation curve for Ni

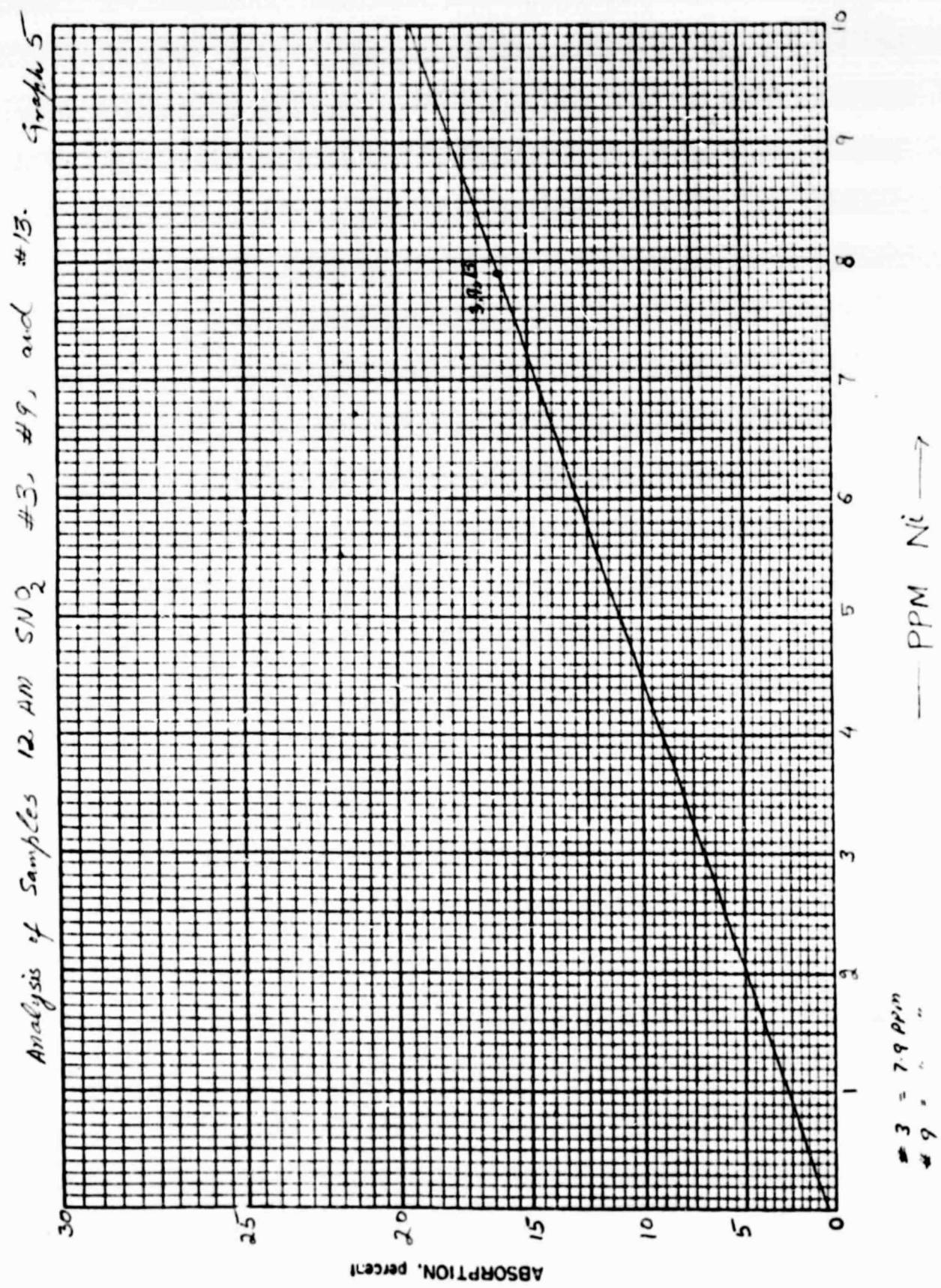
Table IVa

| PPM | A.A. Reading | %Abs |
|-----|--------------|------|
| 2 | .021 | 4.7 |
| 4 | .0398 | 7.8 |
| 6 | .0622 | 13.3 |
| 8 | .0802 | 15.9 |
| 10 | .0920 | 19.2 |

Unknown sample analysis

Table IVb

| Sample No. | Dilution factor | A.A. reading | %Abs | PPM | PPM Orig solution |
|-------------|-----------------|--------------|------|------|-------------------|
| GEO AMP #3 | 250 | .060 | 12.9 | 6.23 | 1557.0 |
| GEO AMP #9 | 250 | .060 | 12.9 | 6.23 | 1557.0 |
| GEO AMP #13 | 250 | .060 | 12.9 | 6.23 | 1557.0 |



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Data for Graph V

Ni analysis of cell 12 AM SNO₂ plates #3, #9, #13

Calibration curve for Ni

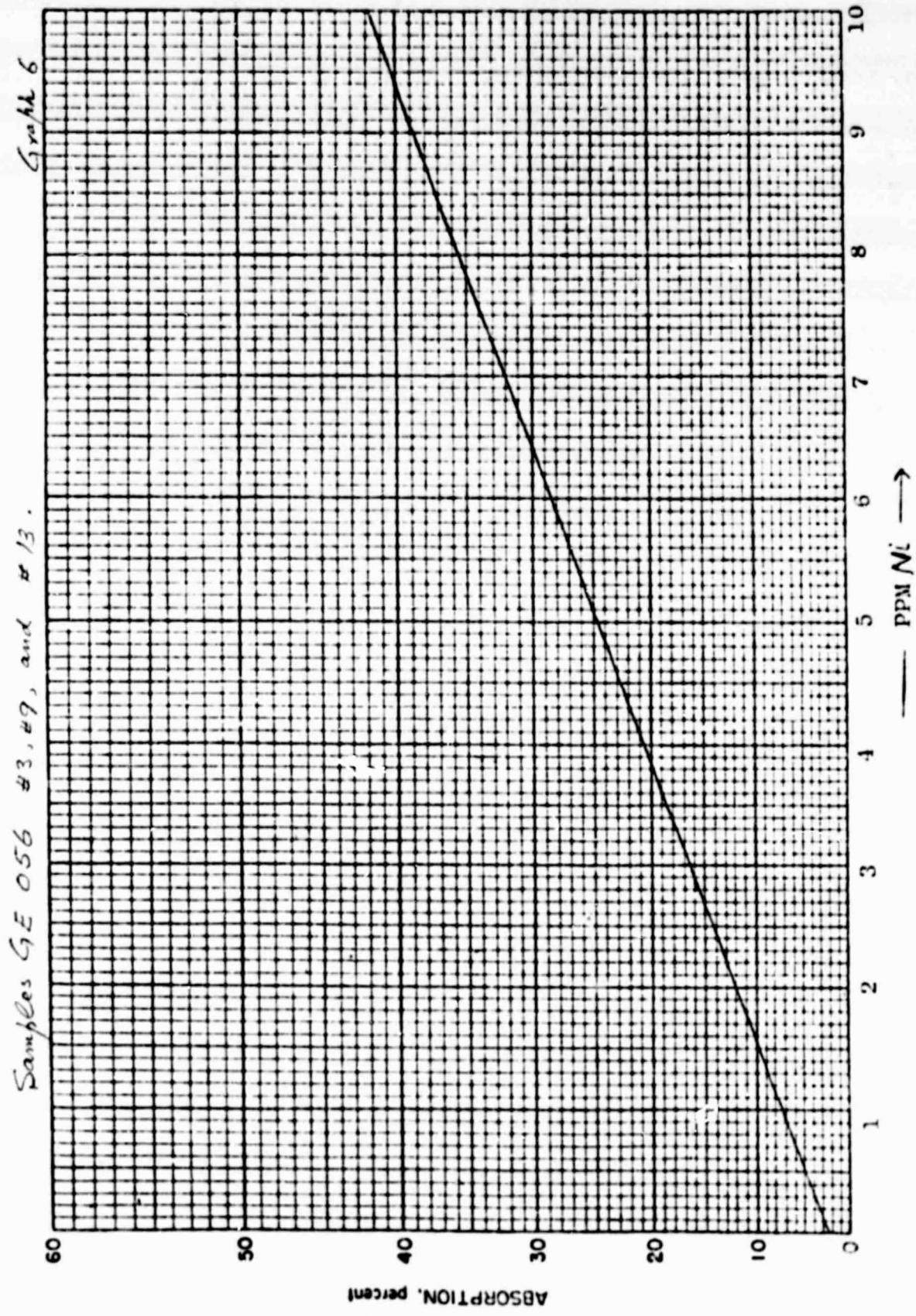
Table Va

| PPM | A.A. Reading | %Abs |
|-----|--------------|------|
| 2 | .022 | 5.0 |
| 4 | .040 | 9.1 |
| 6 | .060 | 13.0 |
| 8 | .079 | 16.7 |
| 10 | .095 | 19.3 |

Unknown sample analysis

Table Vb

| Sample No. | Dilution factor | A.A reading | %Abs | PPM | PPM Orig solution |
|--------------------------|-----------------|-------------|------|-----|-------------------|
| 12AM SNO ₂ #3 | 10 | .077 | 16.3 | 7.9 | 79.0 |
| " #9 | 10 | .080 | 16.5 | 7.9 | 79.0 |
| " | 10 | .078 | 16.4 | 7.9 | 79.0 |



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Data for Graph VI

Ni analysis of cell GE 056 plates #3, #9, #13

Caliberation curve for Ni

Table VIa

| PPM | A.A. Reading | %Abs |
|-----|--------------|------|
| 2 | .053 | 11.5 |
| 4 | .100 | 20.6 |
| 6 | .144 | 28.2 |
| 8 | .190 | 35.4 |
| 10 | .220 | 39.7 |

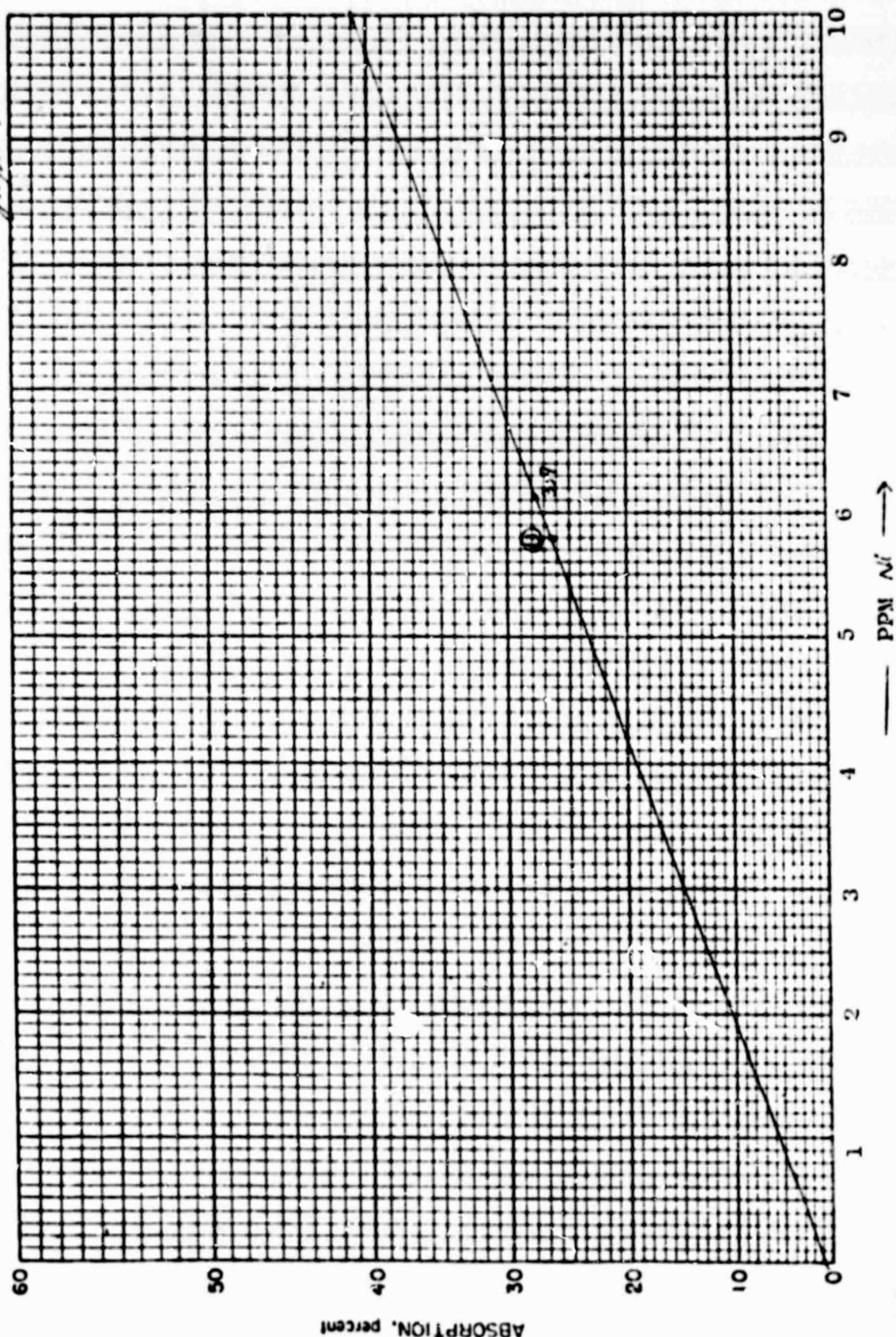
Unknown sample analysis

Table VIb

| Sample No. | Dilution factor | A.A. reading | %Abs | PPM | PPM Orig solution |
|------------|-----------------|--------------|------|------|-------------------|
| GE 056 #3 | 10 | .195 | 36.2 | 8.30 | 83.0 |
| " #9 | " | .198 | 36.6 | 8.42 | 84.2 |
| " #13 | " | .205 | 37.6 | 8.72 | 87.2 |

Samples: GE 056 AM #3, #9, and #13.

Graph 7



3 = 6.13
9 = 6.13
13 = 5.8

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DATA FOR GRAPH VII

Ni analysis of cell GE 056 plates #3, #9, #13, AM Extract

Calibration curve for Ni

Table VIIa

| PPM | A.A. Reading | %Abs |
|-----|--------------|------|
| 2 | .0498 | 10.9 |
| 4 | .0956 | 19.8 |
| 6 | .141 | 27.8 |
| 8 | .182 | 34.3 |
| 10 | .215 | 39.1 |

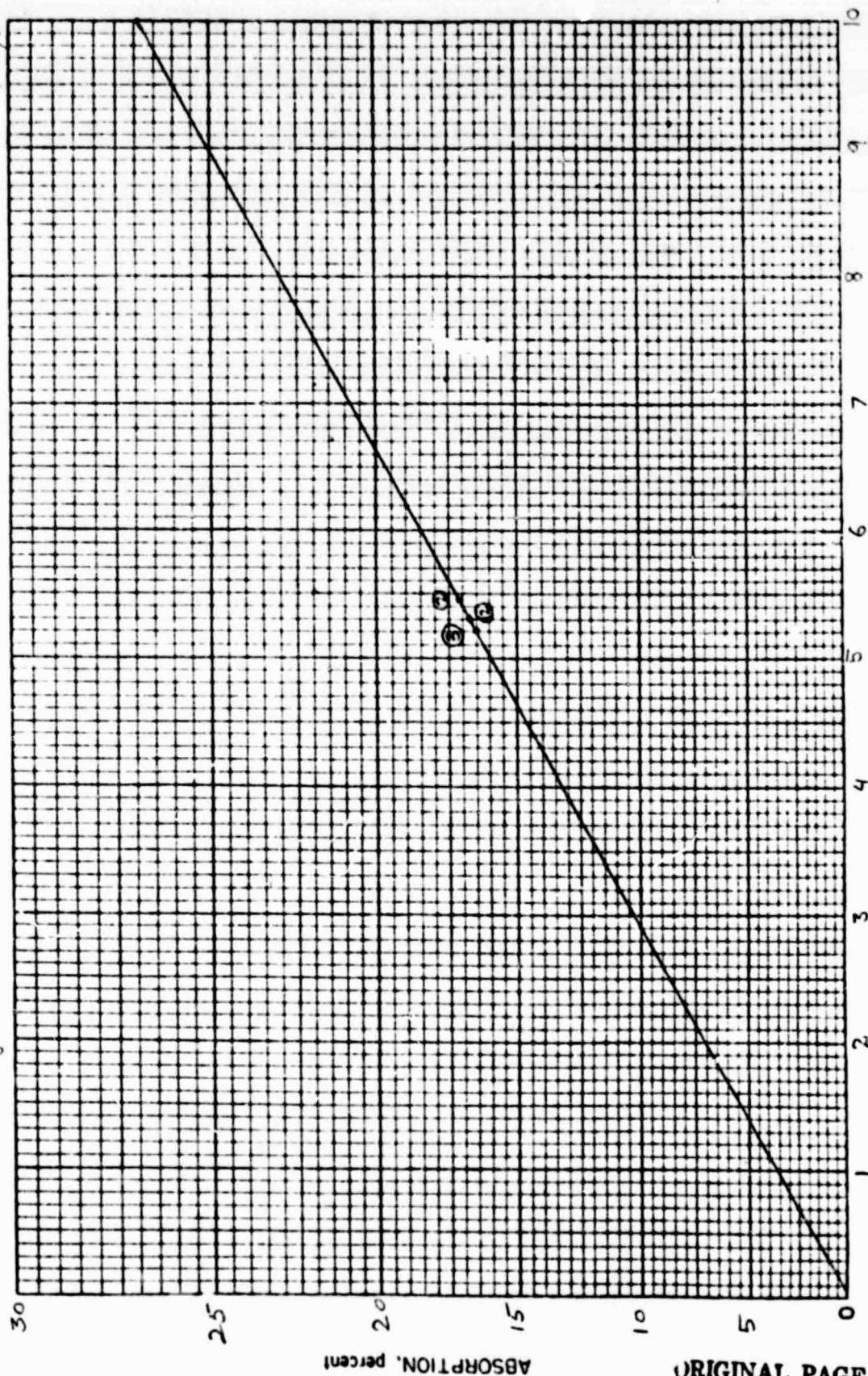
Unknown sample analysis

Table VIIb

| Sample No. | Dilution factor | A.A. reading | %Abs | PPM | PPM Orig solution |
|------------|-----------------|--------------|------|------|-------------------|
| GE 056 #3 | 250 | .142 | 27.9 | 6.13 | 1532.0 |
| " #9 | 250 | .141 | 27.8 | 6.13 | 1532.0 |
| " #13 | 250 | .136 | 26.9 | 5.8 | 1450.0 |

analysis of Samples SN 01 positive #2, #3 and #12.

Graph 8



ABSORPTION, percent

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#3 = 5.2 ppm
#2 = 5.43 " "
#12 = 5.3 "

Data for Graph VIII

Ni analysis of cell SN 01 plates #2, #3, #12

Caliberation curve for Ni

Table VIIIa

| PPM | A.A. Reading | %Abs |
|-----|--------------|------|
| 2 | .031 | 6.8 |
| 4 | .058 | 12.5 |
| 6 | .085 | 17.7 |
| 8 | .112 | 22.8 |
| 10 | .136 | 26.9 |

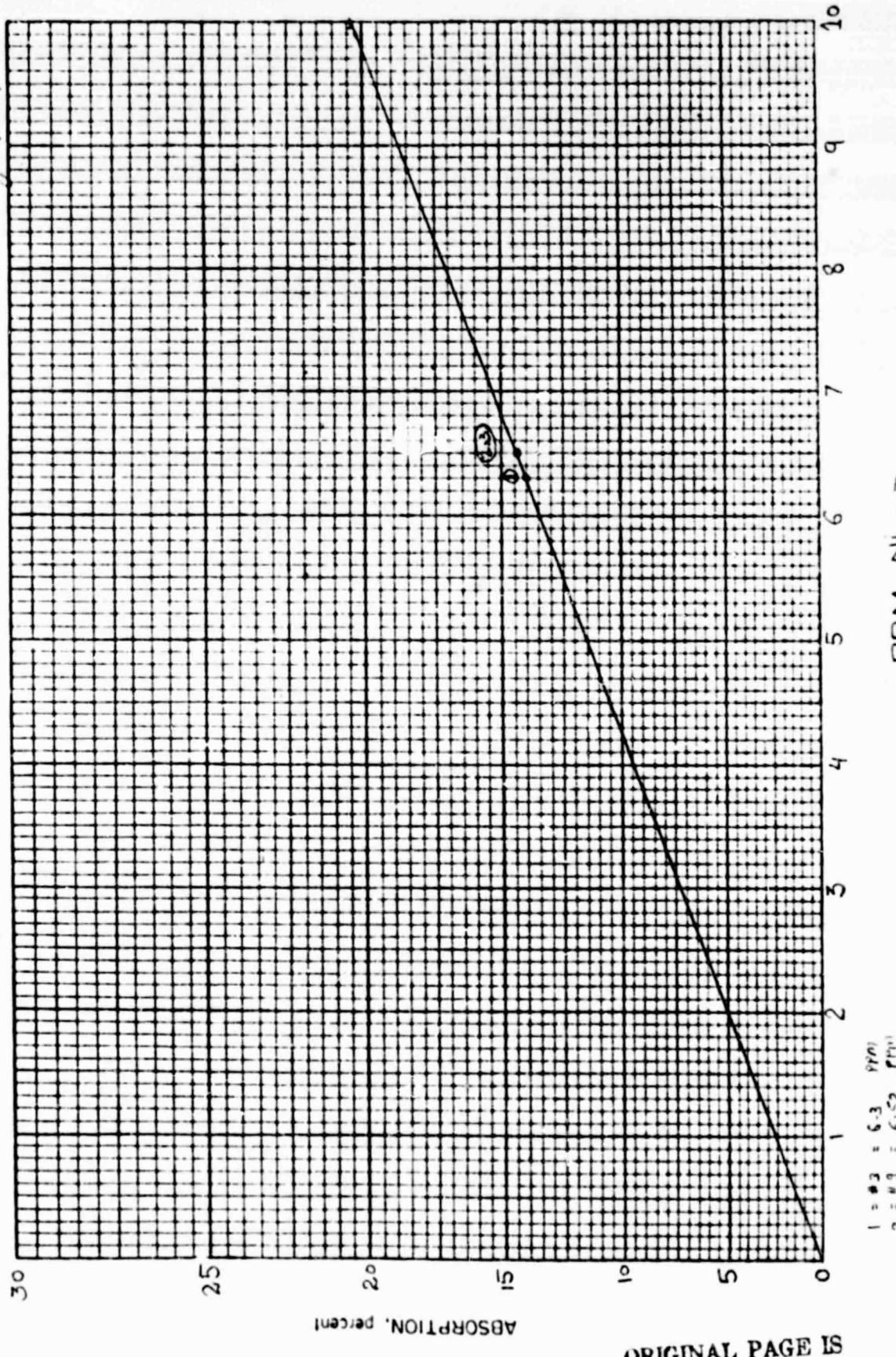
Unknown sample analysis

Table VIIIb

| Sample No. | Dilution factor | A.A. reading | %Abs | PPM | PPM Orig solution |
|------------|-----------------|--------------|------|------|-------------------|
| SN 01 #2 | 250 | .077 | 16.2 | 5.43 | 1357.0 |
| SN 01 #3 | 250 | .073 | 15.5 | 5.2 | 1300.0 |
| SN 01 #12 | 250 | .075 | 15.8 | 5.3 | 1325.0 |

analysis of GE OR #3, #9, and #13.

graph 9



ABSORPTION, percent

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Data for Graph IX

Calibration curve for Ni

Table IXa

| PPM | A.A. Reading | %Abs |
|-----|--------------|------|
| 2 | .023 | 5.1 |
| 4 | .045 | 9.8 |
| 6 | .064 | 13.7 |
| 8 | .083 | 17.3 |
| 10 | .099 | 20.3 |

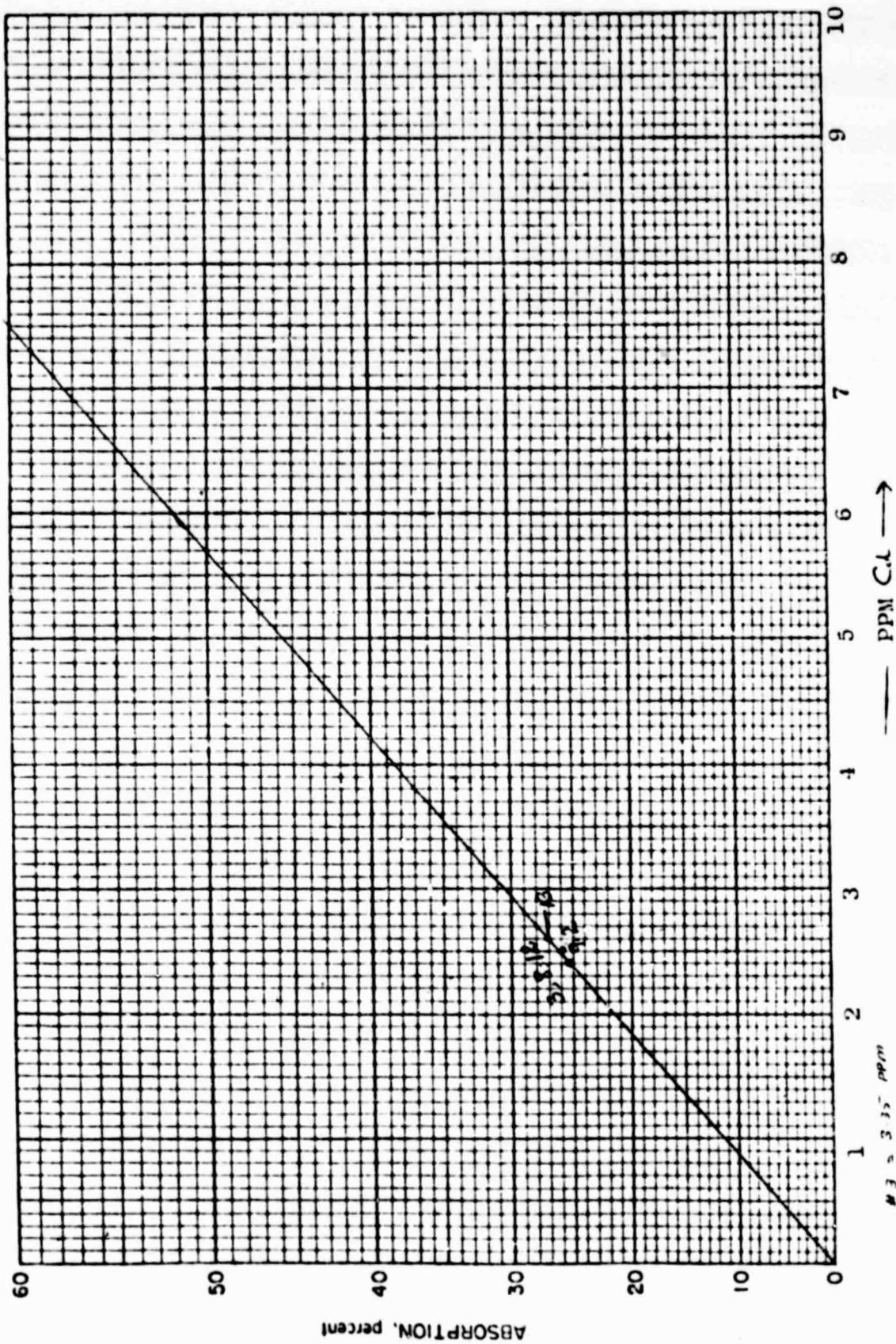
Unknown sample analysis

Table IXb

| Sample No. | Dilution factor | A.A. reading | %Abs | PPM | PPM Orig solution |
|--------------|-----------------|--------------|-------|------|-------------------|
| GE 02 S/N 01 | #3 | 250 | .066 | 14.0 | 6.3 |
| " | #9 | " | .0664 | 14.2 | 6.52 |
| " | #13 | " | .067 | 14.4 | 6.49 |

Analysis of Samples 9E 12 AM SN 02 Negative #3, #9 and #13
Positive #2, #8 and #12

Graph 10



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Data for Graph X

Cd analysis of cell GE 12 AM SN 01

Negative plates #3, #9, #13

Positive plates #2, #8, #12

Caliberation curve for Cd

Table Xa

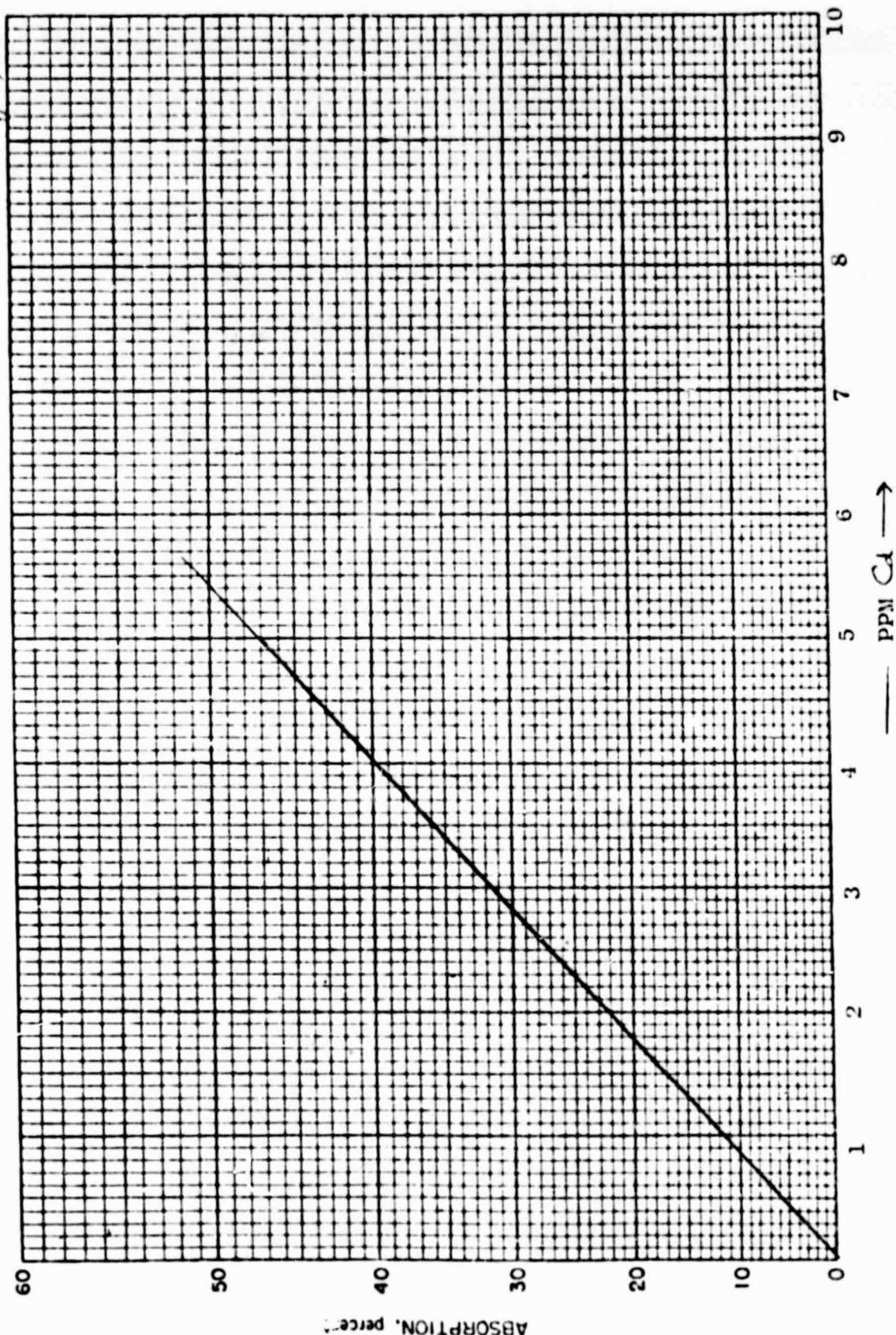
| PPM | A.A. Reading | %Abs |
|-----|--------------|------|
| 1 | .065 | 11.5 |
| 2 | .123 | 24.6 |
| 3 | .174 | 33.0 |
| 4 | .224 | 40.3 |
| 5 | .267 | 46.0 |

Unknown sample analysis

Table Xb

| Sample No. | Dilution factor | A.A. reading | %Abs | PPM | PPM Orig solution |
|---------------|-----------------|--------------|------|------|-------------------|
| GE 12AM SN 01 | 250 | .189 | 35.3 | 3.35 | 837 |
| " | " | .199 | 36.8 | 3.51 | 877 |
| " | " | .202 | 37.2 | 3.60 | 900 |
| " | 50 | .146 | 28.6 | 2.49 | 248 |
| " | " | .144 | 28.2 | 2.41 | 241 |
| " | " | .145 | 28.4 | 2.42 | 242 |

A Calibration Curve of Standard Cu solution to verify reproducibility of Curve Graph 22



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Data for Graph XIII

A run of known Cd solutions was made to make sure for the reproducibility of standard calibration.

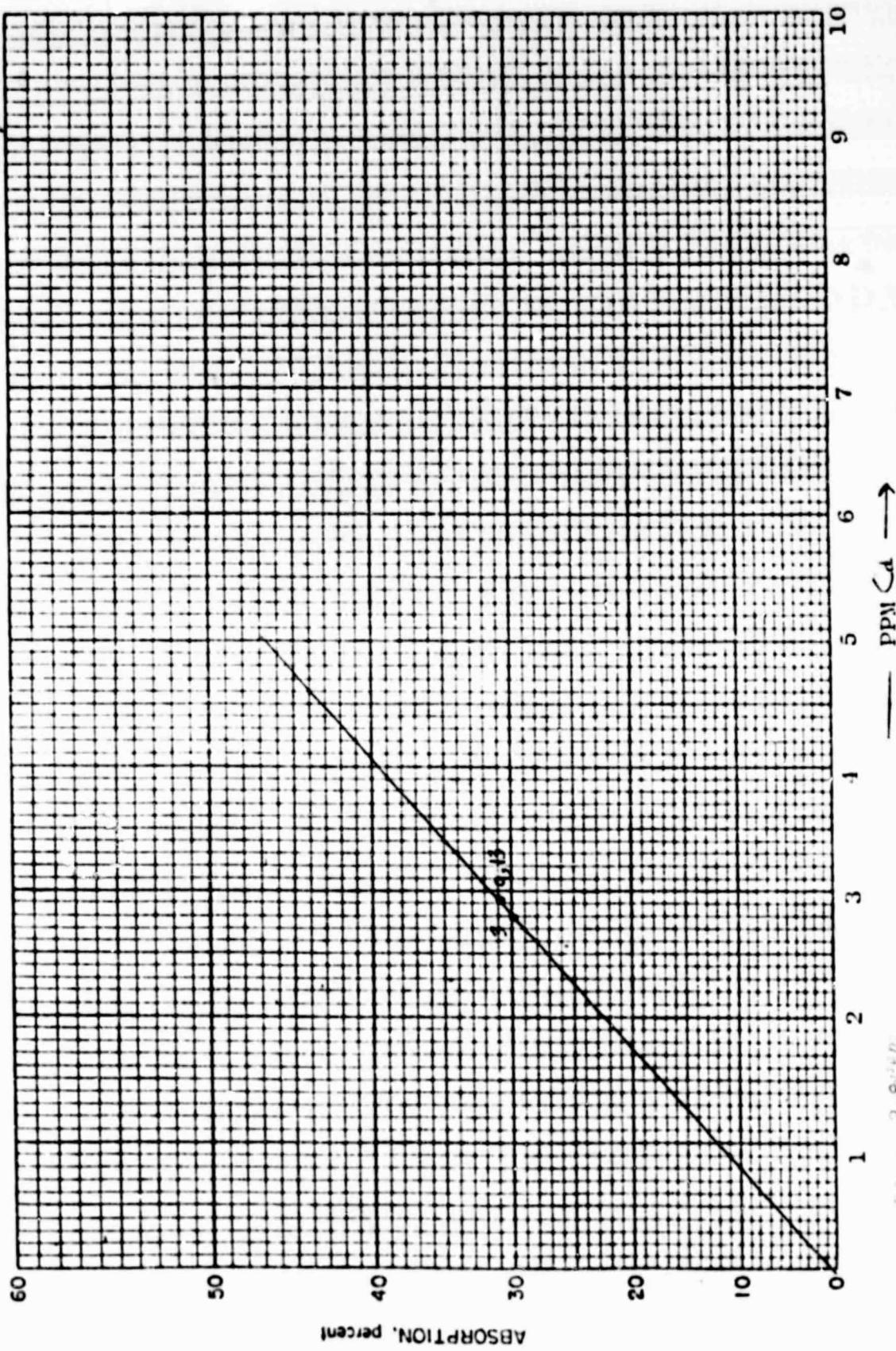
Table XIII, graph 13

| PPM | A.A. Digital Readout | %Abs |
|-----|----------------------|------|
| 1 | .0658 | 14.1 |
| 2 | .1242 | 24.9 |
| 3 | .1766 | 33.4 |
| 4 | .225 | 40.5 |
| 5 | .2672 | 46.0 |

The calibration curve is reproducible.

Samples 12 AM SNO₂ GE 0.2 #3, #7 and #13.

Graph 12



Data for Graph XII

Caliberation curve for Cd

Table XII d

| PPM | A.A. Reading | %Abs |
|-----|--------------|------|
| 1 | .069 | 14.7 |
| 2 | .1276 | 25.5 |
| 3 | .2278 | 33.5 |
| 4 | .2714 | 40.8 |
| 5 | | 46.5 |

Unknown sample analysis

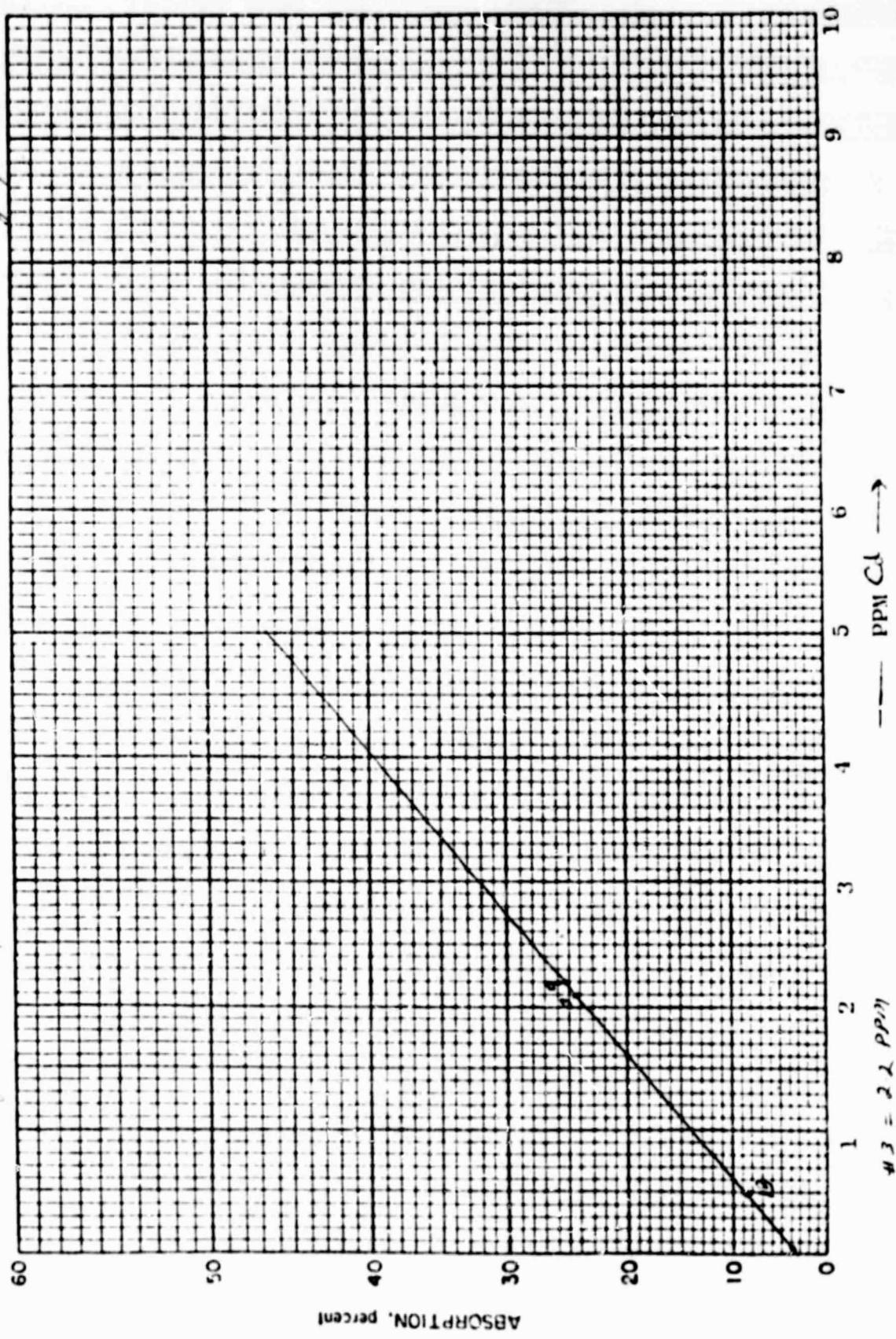
Table XII b

| Sample No. | Dilution factor | A.A. reading | %Abs | PPM | PPM Orig solution |
|---------------|-----------------|--------------|------|------|-------------------|
| 12AM GE 02 #3 | 250 | .1676 | 32.0 | 2.8 | 700 |
| " " #9 | 250 | .1744 | 33.1 | 2.9 | 725 |
| " " #13 | 250 | .1756 | 33.3 | 2.93 | 732.5 |

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Graph 13

Analysis of Samples 4E 02 AM, P #3, #7, and #13.



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Data for Graph X III

Cd analysis of cell GE 02 plates #3, #9, #13

Caliberation curve for Cd

Table X IIIa

| PPM | A.A. Reading | %Abs |
|-----|--------------|------|
| 1 | .0646 | 13.8 |
| 2 | .1256 | 25.2 |
| 3 | .1764 | 33.4 |
| 4 | .2244 | 40.4 |
| 5 | .2738 | 45.4 |

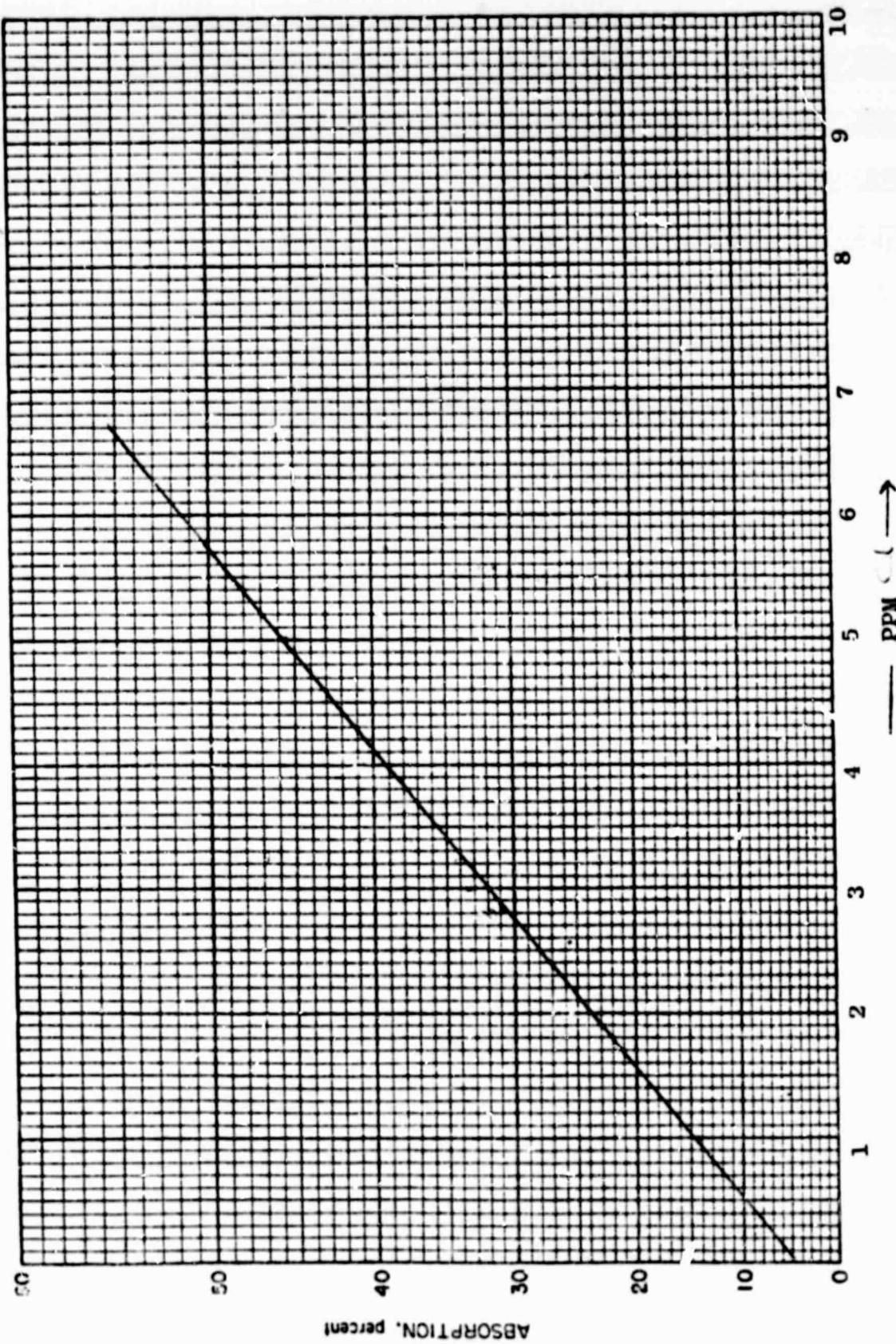
Unknown sample analysis

Table X IIIb

| Sample No. | Dilution factor | A.A. reading | %Abs | PPM | PPM Orig solution |
|------------|-----------------|--------------|------|------|-------------------|
| GE 02 #3 | 0 | .1356 | 26.8 | 2.20 | 2.20 |
| " #9 | 0 | .123 | 24.7 | 2.10 | |
| " #13 | 0 | .0322 | 7.1 | 0.5 | 0.50 |

Analyses of samples 12AN 5NO₂ 6E 02 23, 7, 13.

Graph 14



3: 2.6 ppm
7: 3.9
13: 2.7

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Data for Graph XIV

Cd analysis of cell 12 AH SNO₂ GE02 plates #3, #9, #13.

Calibration curve for Cd

Table XIVa

| PPM | A.A. Reading | %Abs |
|-----|--------------|------|
| 1 | .069 | 14.7 |
| 2 | .128 | 25.5 |
| 3 | .177 | 33.5 |
| 4 | .228 | 40.8 |
| 5 | .271 | 46.5 |

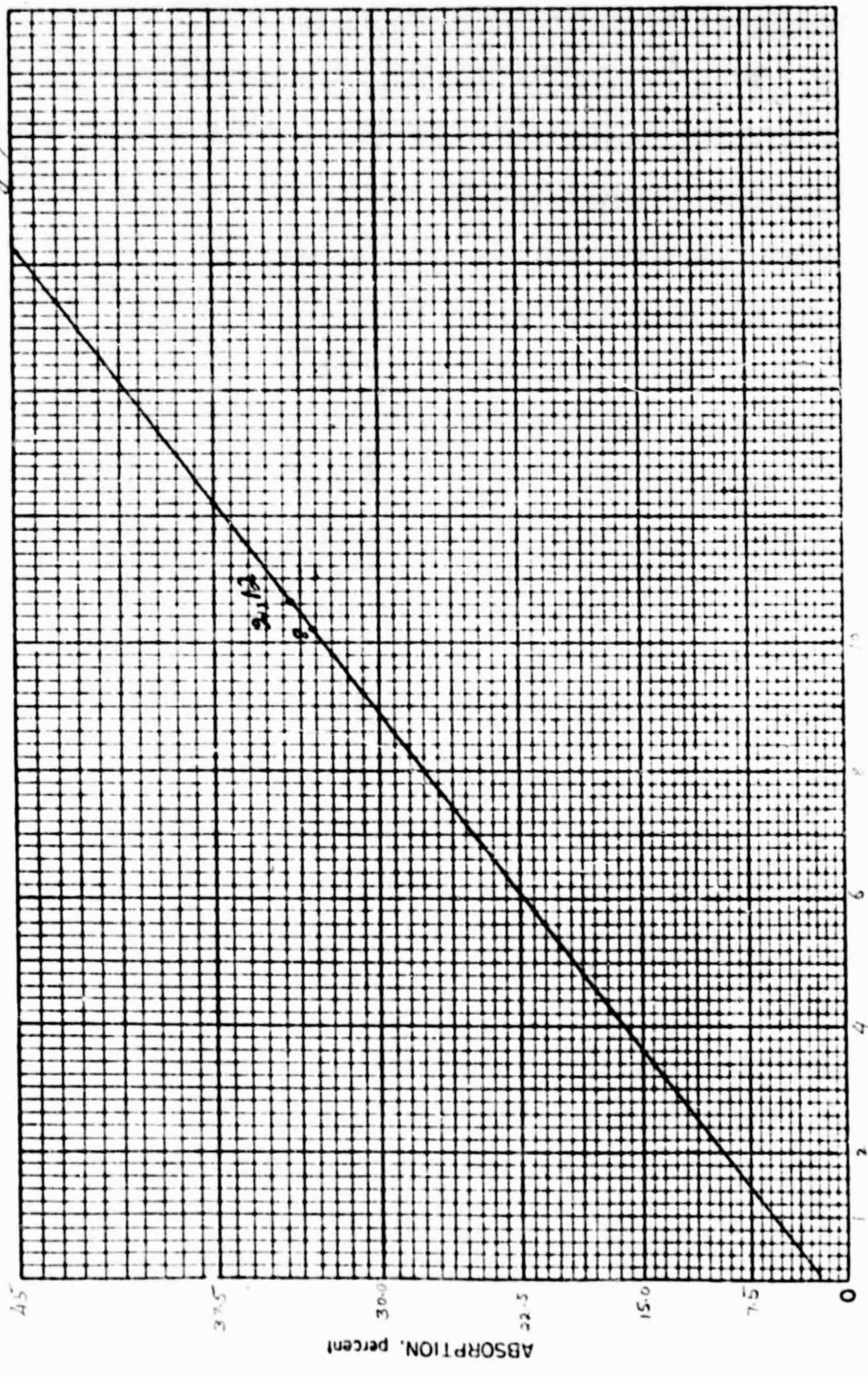
Unknown sample analysis

Table XIVb

| Sample No. | Dilution factor | A.A. reading | %Abs | PPM | PPM Orig solution |
|------------------------------|-----------------|--------------|------|------|-------------------|
| 12 AH SNO₂ | | | | | |
| GE 02 | #3 | 250 | .168 | 32.0 | 2.80 |
| " | #9 | " | .174 | 33.1 | 2.90 |
| " | #13 | " | .176 | 33.3 | 2.93 |

Cold finger extract GE 056 #2, #8, and #12.

graph 15



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Data for Graph XY

Cobalt analysis of cell GE 056 plates, #2, #8,

Calibration curve for Co

Table Xla

| PPM | A.A. Reading | %Abs |
|-----|--------------|------|
| 1 | .020 | 4.5 |
| 2.5 | .051 | 11.1 |
| 4 | .078 | 16.5 |
| 5 | .096 | 19.9 |
| 6 | .113 | 22.9 |
| 8 | .1465 | 28.6 |
| 10 | .173 | 32.9 |

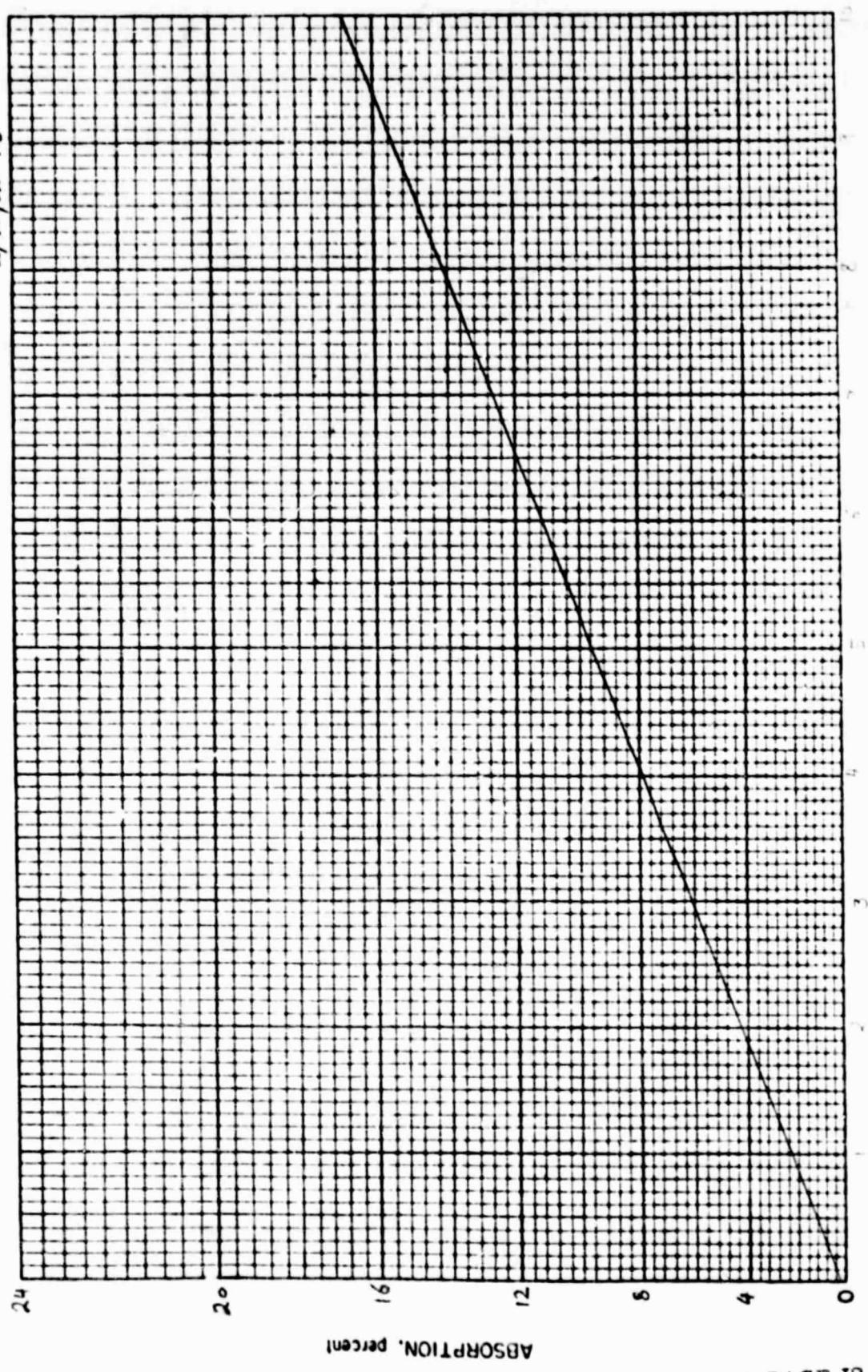
Unknown sample analysis

Table Xlb

| Sample No. | Dilution factor | A.A. reading | %Abs | PPM | PPM Orig solution |
|---------------|--------------------|-----------------|------|-------|----------------------|
| GE 056 #2 | 5 | .183 | 34.4 | 10.35 | 52.0 |
| " #8 | 5 | .179 | 33.8 | 10.2 | 51.0 |
| " #12 | 5 | .181 | 34.1 | 10.3 | 52.0 |

Sample GE 02 #2 8/12.

Graph 16



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Data for Graph XVI

Co analysis of cell GE 02 Positive plates #2, #8, #12

Calibration curve for Co

Table XVIa

| PPM | A.A. Reading | %Abs |
|-----|--------------|------|
| 1 | .010 | 2.3 |
| 2.5 | .023 | 5.2 |
| 4 | .036 | 8.0 |
| 5 | .043 | 9.5 |
| 6 | .051 | 11.1 |
| 8 | .067 | 14.3 |
| 10 | .081 | 17 |

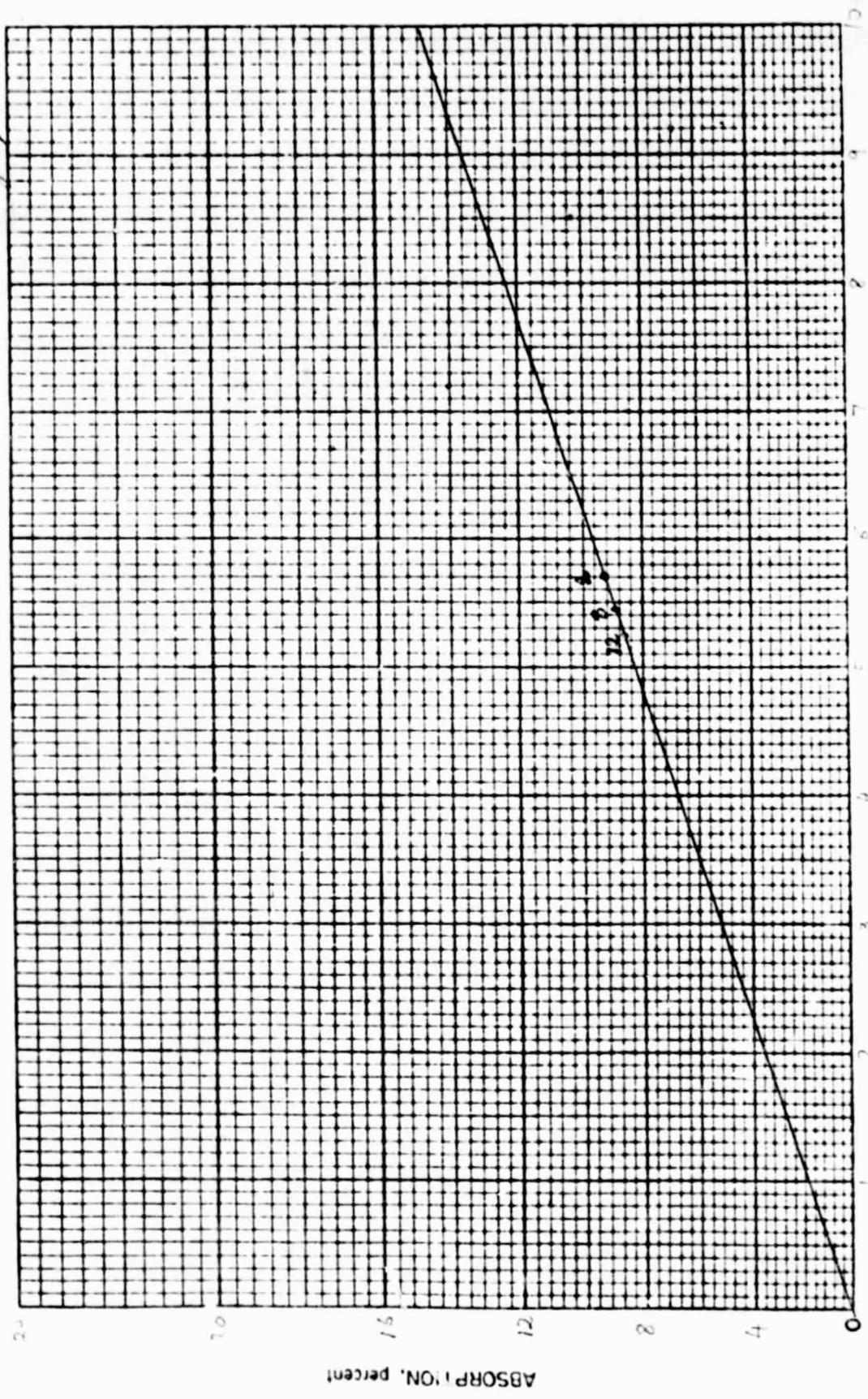
Unknown sample analysis

Table XVIb

| Sample No. | Dilution factor | A.A. reading | %Abs | PPM | PPM Orig solution |
|------------|-----------------|--------------|------|-------|-------------------|
| GE 02 #2 | 5 | .083 | 17.4 | 10.15 | 51.0 |
| " #8 | 5 | .086 | 17.8 | 10.35 | 52.0 |
| " #12 | 5 | .081 | 17.0 | 10.00 | 50.0 |

Logistics of SE 12 SN 02 #2, 8, 12.

Maple 17



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2 = 5.7
8 = 5.45
12 = 5.25

Data for Graph X

Co analysis of cell GE 12 SN/01 plates #2, #8, #12

Calibration curve for Co

Table XVII-a

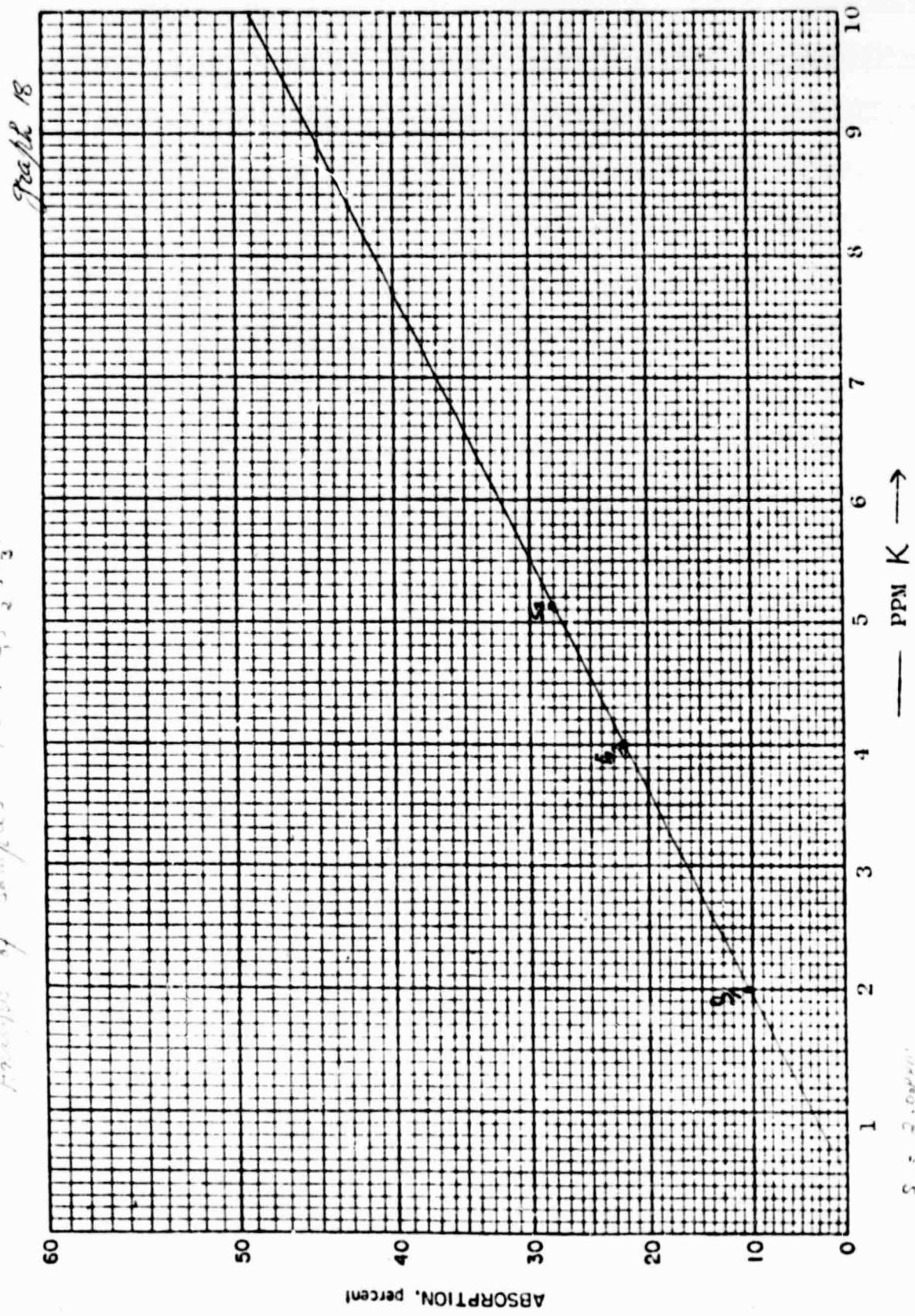
| PPM | A.A. Reading | %Abs |
|-----|--------------|------|
| 1 | .007 | 1.6 |
| 2.5 | .019 | 4.3 |
| 4 | .030 | 6.7 |
| 5 | .037 | 8.2 |
| 6 | .044 | 9.6 |
| 8 | .058 | 12.5 |
| 10 | .070 | 14.9 |

Unknown sample analysis

Table XVII-b

| Sample No. | Dilution factor | A.A. reading | %Abs | PPM | PPM Orig solution |
|---------------|-----------------|--------------|------|------|-------------------|
| GE 12SN 01 #2 | 5 | .042 | 9.2 | 5.7 | 28.5 |
| " 11SN 01 #8 | 5 | .040 | 8.8 | 5.45 | 27.25 |
| " " " #12 | 5 | .039 | 8.6 | 5.25 | 26.25 |

Percentages of Samples P.O.T., S₁, S₂, S₃



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Data for Graph XVIII

K Analysis of plates S₁, S₂, and S₃

Calibration curve for K

Table XVIIa

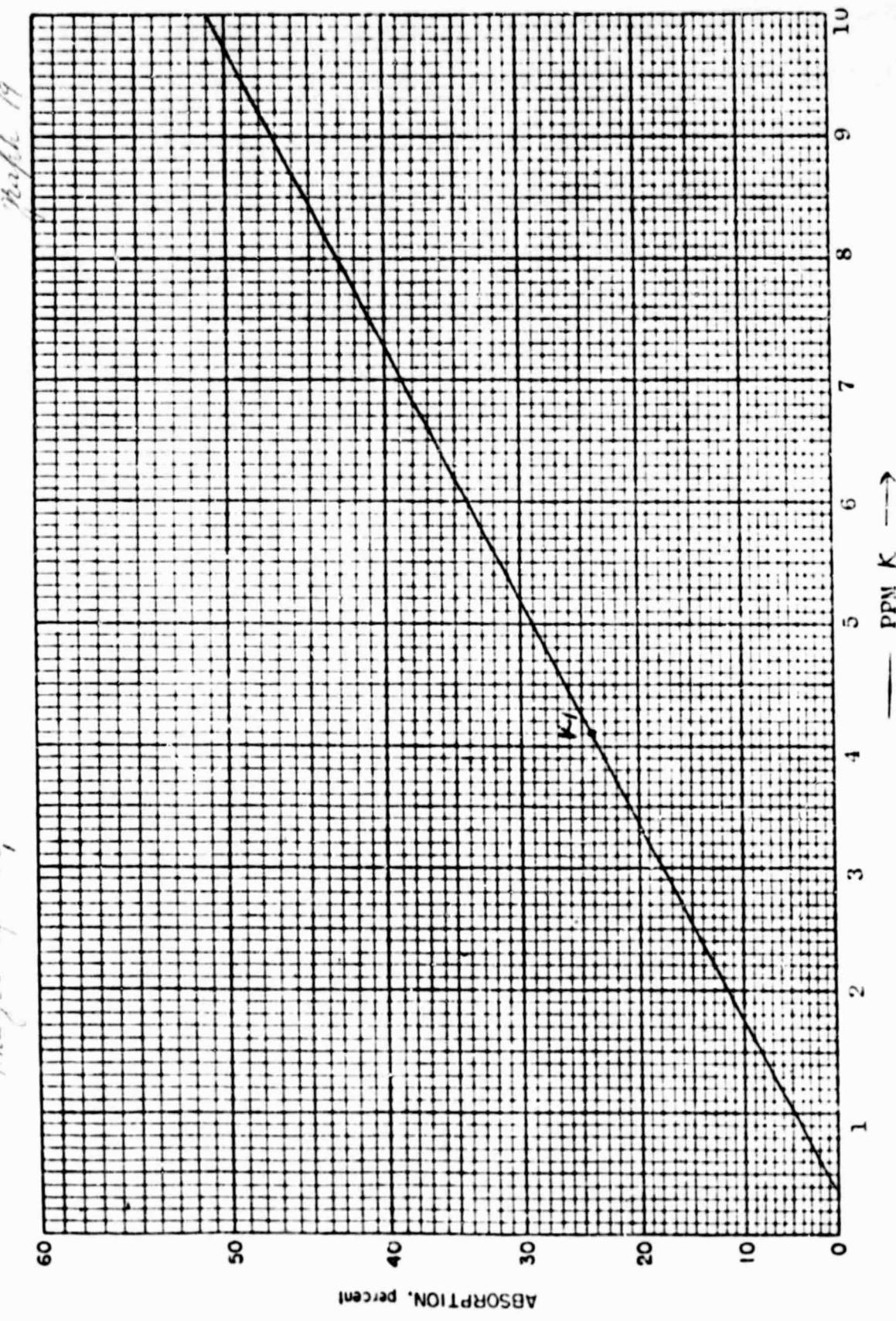
| PPM | A.A. - Reading | %Abs |
|-----|----------------|------|
| 1 | .024 | 5.4 |
| 2 | .0488 | 10.6 |
| 4 | .1066 | 21.8 |
| 6 | .1734 | 32.9 |
| 8 | .2436 | 42.9 |
| 10 | .3202 | 52.2 |

Unknown sample analysis

Table XVIIIb

| Sample No. | Dilution factor | A.A. reading | %Abs | PPM | PPM Orig solution |
|----------------|-----------------|--------------|------|------|-------------------|
| S ₁ | 0 | .0492 | 10.7 | 2.0 | 2.0 |
| S ₂ | 0 | .107 | 21.8 | 4.0 | 2.0 |
| S ₃ | 2 | .1458 | 28.5 | 5.15 | 10.30 |

Analysis of K_1



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$K_1 = 4.7 \text{ ppm}$

Data for Graph XIX

Calibration curve for K

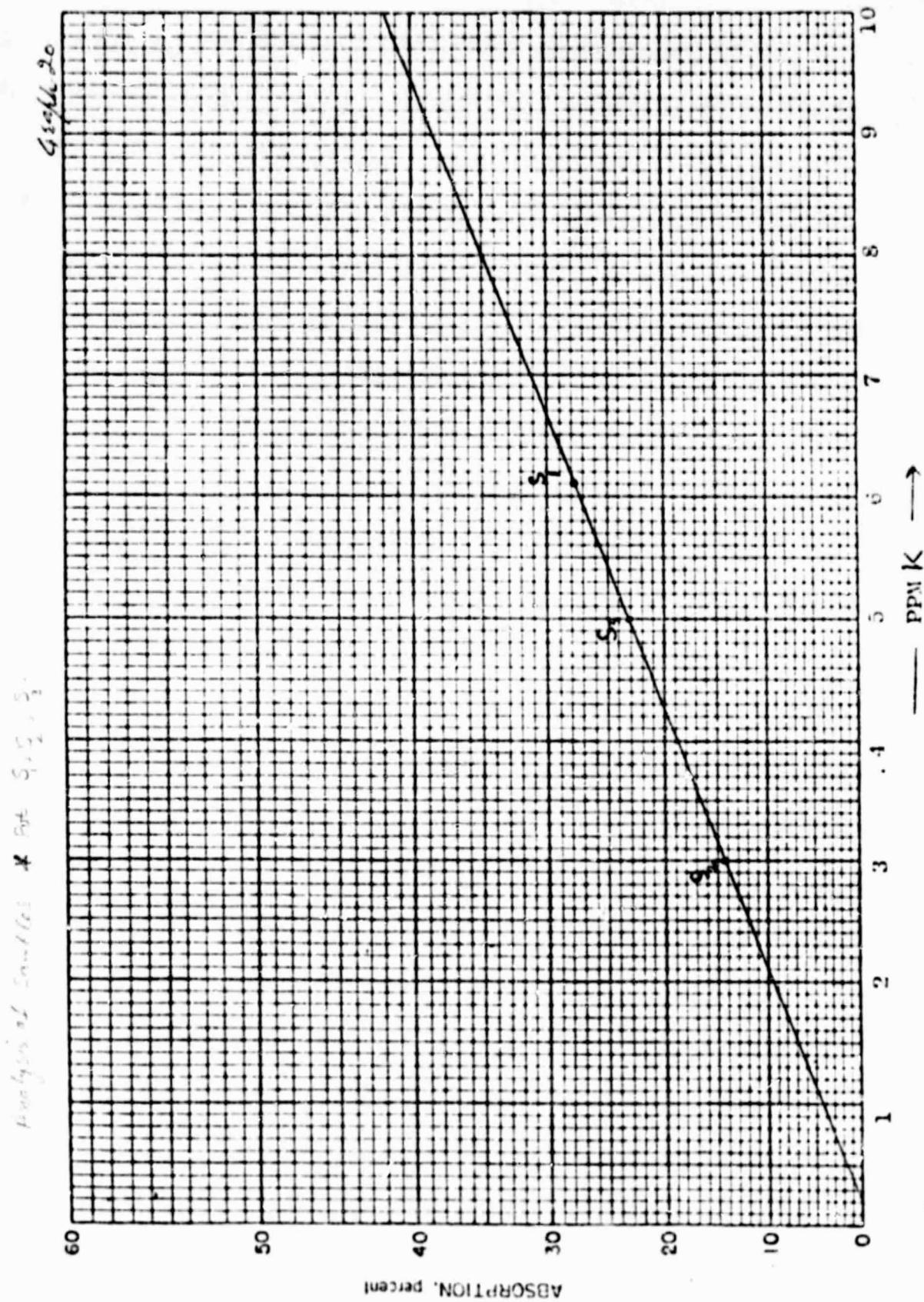
Table XIXa

| PPM | A.A. Reading | %Abs |
|-----|--------------|------|
| 1 | .028 | 6.30 |
| 2 | .063 | 13.5 |
| 4 | .108 | 22.1 |
| 6 | .178 | 33.7 |
| 8 | .257 | 44.6 |
| 10 | .333 | 53.6 |

Unknown sample analysis

Table XIXb

| Sample No. | Dilution factor | A.A. reading | %Abs | PPM | PPM Orig solution |
|----------------|-----------------|--------------|------|-----|-------------------|
| K ₁ | 250 | .114 | 23.1 | 4.1 | 1025.0 |



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Data for Graph XX

K analysis of plates S₁, S₂, S₃

Calibration curve for K

Table XXa

| PPM | A.A. - Reading | %Abs |
|-----|----------------|------|
| 1 | .0138 | 3.1 |
| 2 | .0262 | 5.8 |
| 3 | .0450 | 9.8 |
| 4 | .0666 | 14.2 |
| 5 | .0930 | 19.3 |
| 6 | .199 | 24.0 |
| 7 | .150 | 29.2 |
| 8 | .176 | 33.3 |
| 9 | .208 | 38.1 |
| 10 | .234 | 41.8 |

Unknown sample analysis

Table XXb

| Sample No. | Dilution factor | A.A. reading | %Abs | PPM | PPM Orig solution |
|----------------|-----------------|--------------|------|-----|-------------------|
| S ₁ | 10 | .121 | 24.3 | 6.1 | 61.0 |
| S ₂ | " | .048 | 10.4 | 3.0 | 30.0 |
| S ₃ | " | .093 | 19.3 | 5.0 | 50.0 |

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