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Thermo Electron Corporation, 85 First Avenue, Waltham, Massachusetts 02154

Report No. TE 4092/3-53-69

THIRD QUARTERLY PROGRESS REPORT
APPLIED THERMIONIC RESEARCH

Contract 952184

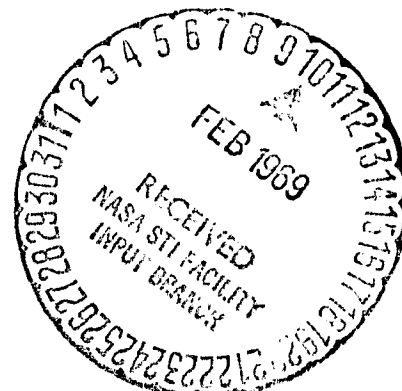
4 June 1968 to 4 November 1968

D. Lieb, F. Rufeh, M. Shaw
and L. van Someren

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Pasadena, California

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TABLE OF CONTENTS

<u>Chapter</u>		<u>Page</u>
I	INTRODUCTION AND SUMMARY	I-1
II	MASS SPECTROMETRIC INVESTIGATION OF ELECTRONEGATIVE ADDITIVES	II-1
	A. Introduction	II-1
	B. Preliminary Modifications and Manipulations .	II-1
	C. Cesium Oxide Experimentation	II-1
	1. First Run	II-3
	2. Second Run	II-3
	D. Conclusions	II-4
III	PARAMETRIC DATA	III-1
	A. Emission Characteristics of a Duplex Vapor- Deposited Emitter	III-1
	1. Introduction	III-1
	2. Emitter Preparation	III-1
	3. Work Function Measurements	III-2
	4. Cesium Families	III-3
	5. Spacing Families	III-3
	6. Summary of the Data	III-3
	7. Correlations	III-4
	B. Vacuum Work Function Measurement Apparatus	III-6
	References	III-7



CHAPTER I

INTRODUCTION AND SUMMARY

The mass spectrometric system for the investigation of electro-negative additives was modified and data were obtained from a cesium oxide capsule at temperatures up to 240°C.

The emission characteristics of a vapor-deposited tungsten emitter with a molybdenum collector were investigated in a variable-spacing converter. The set of data obtained is particularly useful for comparison with the results of theoretical analysis and for studies of interrelationships between the various converter parameters, since it includes work function measurements and variable spacing data as well as cesium pressure families. The data were used to investigate the dependence of the voltage drop on the product of cesium pressure and interelectrode spacing Pd , cesium pressure P and ion richness β . At constant values of Pd and β , a dependence on cesium pressure was observed.

The vacuum work function measurement apparatus described in the Second Quarterly report was completed and preliminary data were obtained on a fluoride vapor-deposited emitter.



CHAPTER II

MASS SPECTROMETRIC INVESTIGATION OF ELECTRONEGATIVE ADDITIVES

Marion Shaw

A. INTRODUCTION

During the third quarter, minor modifications on the system were carried out, stability of response of the mass spectrometer was checked out, and a lock-in amplifier system was set up for use. Experimentation was begun on cesium oxides. Data were obtained from one capsule at temperatures up to 240°C.

B. PRELIMINARY MODIFICATIONS AND MANIPULATIONS

The bellows and a heater on the Knudsen cell were replaced. The ion pump surrounding the Knudsen cell was cleaned out, due to the presence of some contamination from the cesium run.

The lock-in amplifier (PAR model 120) was wired up so that the output of the multiplier on the mass spectrometer could be switched either directly into the scope, into the lock-in amplifier, or into the lock-in amplifier preceded by a preamplifier (PAR model 112). The system was tried out with the nitrogen beam apparatus (Figure II-1). The nitrogen beam was also used to verify that the response of the mass spectrometer had not fallen off.

C. CESIUM OXIDE EXPERIMENTATION

A total of three capsules of cesium oxide were built. The first developed a leak in the fuse braze, apparently due to a porous structure caused by hydrogen brazing. Subsequent capsules were vacuum-brazed and the problem was not encountered again. The second capsule apparently



could not be cracked in the Knudsen cell under vacuum, and it was removed. Everything went well with the third capsule and we were able to obtain data from it; the following discussion concerns this capsule.

With the apparatus for distillation and oxidation of cesium shown in Figure II-2, a capsule containing equal stoichiometric amounts of cesium and oxygen (to form CsO) was built. The initial amount of cesium was 1/2 gm. This capsule was placed in the Knudsen cell, and the system with the Knudsen cell was baked out up to a Knudsen cell temperature of 250°C. With the system at room temperature the background pressures were

$$p_1 = 2 \times 10^{-8} \text{ torr (chamber containing Knudsen cell)}$$

$$p_2 = 1 \times 10^{-7} \text{ torr (chamber containing mass spectrometer)}$$

The capsule was cracked and mass scans were taken as a function of the temperature of the Knudsen cell. Mass scans showed that the only modulated signal present (i. e. , from the chopped molecular beam) was due to cesium.

Using the pre-amp, the minimum signal level due to noise which could be observed for this capsule corresponded to about

$$5 \times 10^{-6} \text{ torr - in mass/charge range up to 50}$$

$$5 \times 10^{-5} \text{ torr - in mass/charge range from 50 to 500}$$

The noise was a function of mass setting; the background was much worse at the lower mass numbers.



Both the lock-in amplifier and the scope were observed to track together where the signal was large enough to be seen on the scope. Two runs (described in detail below) from room temperature up to 230°C were taken. Above 230°C the outgassing level was too high for the ion pump. At the end of the second run, the ion pump surrounding the Knudsen cell showed evidence of contamination, and no further data were taken on this capsule.

1. First Run

The results are shown by curve A of Figure II-3. The shape of the curve is qualitatively the same as that found in the second run. The difference in amplitude of a factor of about two between the two runs can be explained at least in part by the fact that better resolution (resulting in lowered intensity) was used in the first run. The signal was qualitatively reproducible in the sense that the curves in Runs I and II had the same shape and the same magnitude within a factor of 2.

2. Second Run

The results are shown by curve B of Figure II-3. It would have been interesting to have followed the cooling curve all the way back to room temperature. At this point, however, the ion pump started drawing too much current. There appeared to be a small peak at 200°C; however, the system was less stable due to outgassing at higher temperatures, and this peak may be an artifact.



D. CONCLUSIONS

Although only cesium was observed, the temperature dependence of the signal was very different from that to be expected from a pure cesium reservoir:

- (1) the temperature dependence was not exponential;
- (2) the magnitude at higher temperatures was much smaller than would be expected for pure cesium.

For example, the signal from pure cesium was observed to be 48 mV at 130°C and 84 mV at 140°C (see second quarterly report). In this experiment with cesium oxide, the maximum cesium signal was 20 mV (145°C) and at 200°C was less than 5 mV. The signal predicted from our data for pure cesium at 200°C would be over 600 mV. According to our calibration, a partial pressure of 1μ of cesium in the Knudsen cell corresponds to a 28 mV signal on the scope.

No other mass peaks at the modulation frequency were observed over the mass/charge range of 1 - 500.

Questions which arise from these data include:

- (1) Can the noise level of the system be reduced so that higher gain can be used in a search for oxides and oxygen?
- (2) Were the contents of the capsule at equilibrium?

We think that the noise level of the system can be significantly improved by baking out to higher temperatures. Also, it is important from the point of view of the converter application to be able to take data as high as 600°C. We think that bakeout at higher temperatures before breaking the capsule will improve the probability that the contents are in an equilibrium state if the oxidation happens to be kinetically limited.

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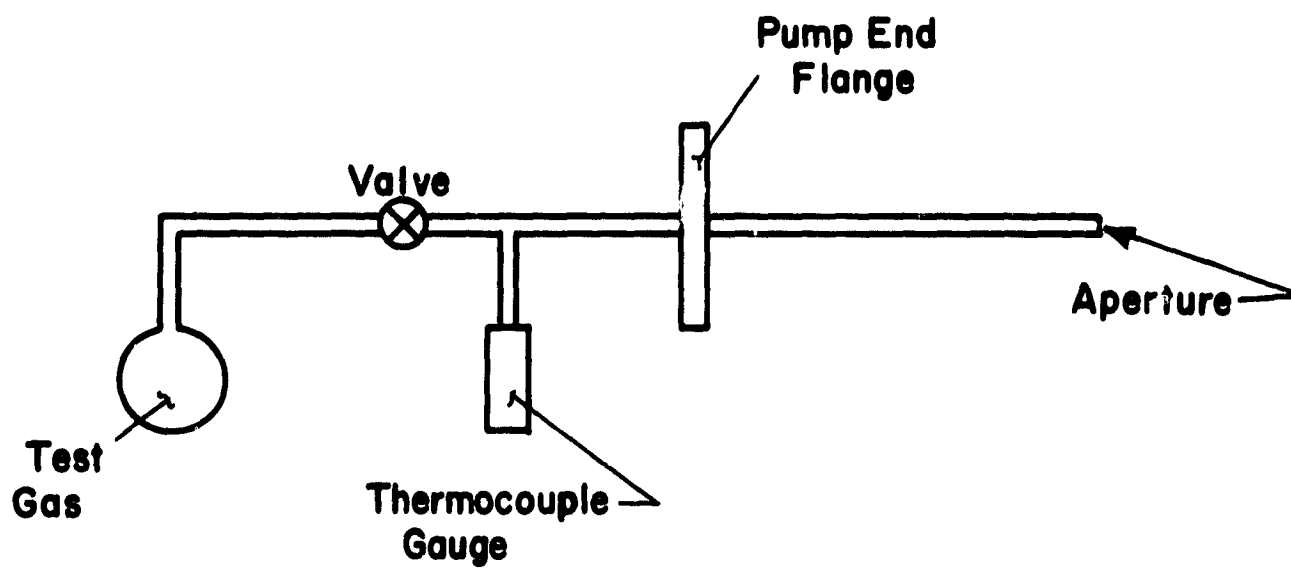


Figure II-1 Gas Manifold.

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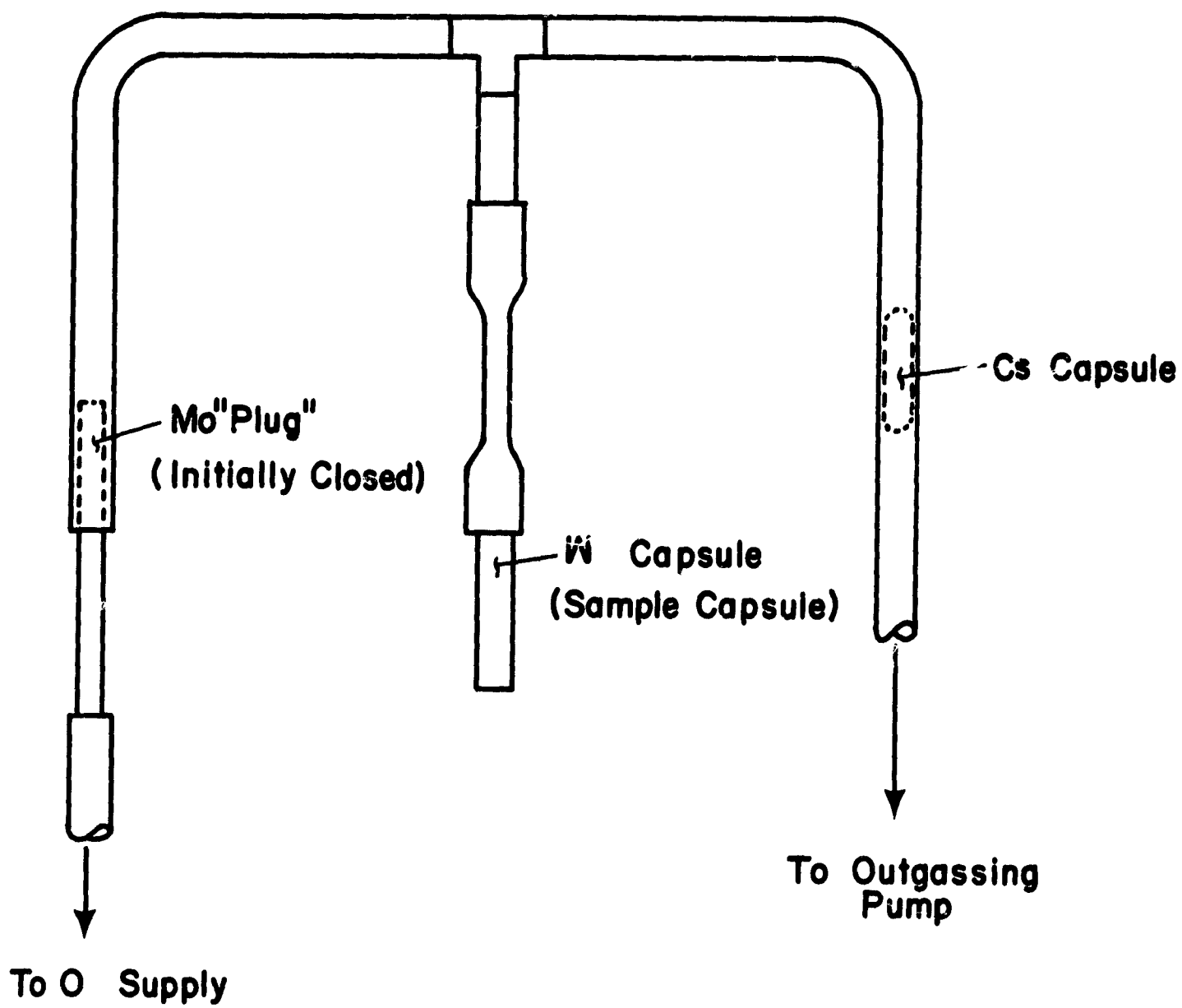


Figure II-2 Cesium Oxide Forming Apparatus.

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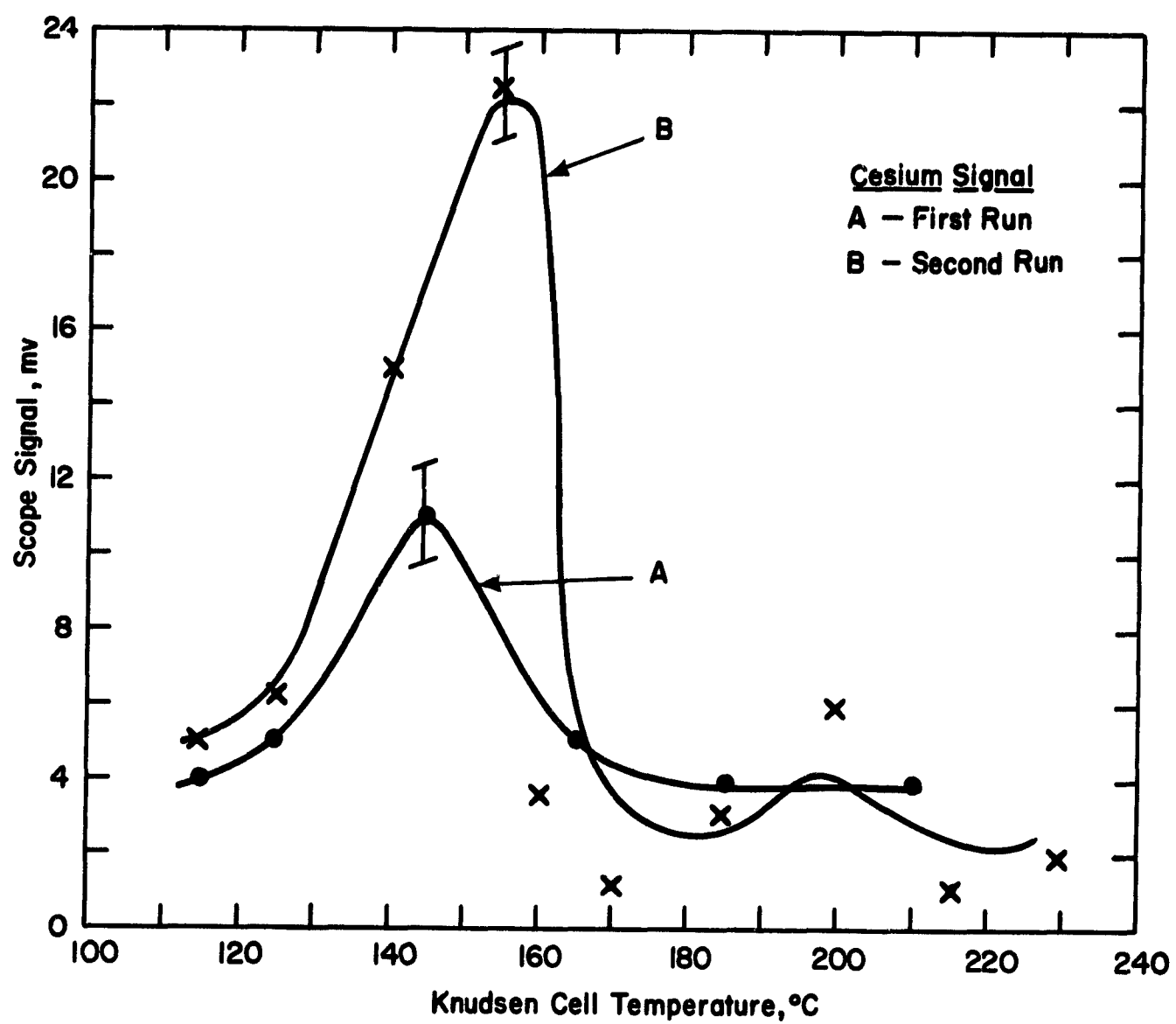


Figure II-3. Mass Spectrometric Output as a Function of Knudsen Cell Temperature, Cs^+ Peak.



CHAPTER III

PARAMETRIC DATA

F. Rufeh, D. Lieb and L. van Someren

A. EMISSION CHARACTERISTICS OF A DUPLEX VAPOR-DEPOSITED EMITTER

1. Introduction

The emission characteristics of a vapor-deposited tungsten emitter with a molybdenum collector were investigated in a variable spacing converter. The converter is equipped with a collector guard ring which is maintained at the same temperature and potential as the collector (Figure III-1). Flexible bellows allow the interelectrode spacing to be varied from zero to 100 mils. The set of data obtained is particularly useful for comparison with the results of theoretical analysis and for studies of the interrelationships between the various converter parameters, since it includes work function measurements and variable spacing families as well as cesium pressure families.

2. Emitter Preparation

The raw material consisted of a piece of fluoride vapor-deposited tungsten from an ingot previously used on this program. A slab was ground down to about 0.155 inch thick, and then a layer of tungsten about 0.02 inch thick was vapor-deposited onto it from the chloride and its surface was ground. The result was to give an emitter with the strength and grain stability of fluoride tungsten combined with the superior emission properties expected from (110) oriented chloride tungsten.



Discs 0.77 inch in diameter were cut from the raw material, and annealed for 3 hours at 2220°K in a tungsten container in a resistance furnace. This was expected to produce a grain structure which would be stable during subsequent converter operation. To ensure removal of the randomly oriented layer introduced by grinding, the emitter face of the disc was electropolished in 5% NaOH solution at 10 volts for two minutes.

An attempt was made to obtain a partial pole figure from the surface of the emitter, but the highly irregular curve which resulted was an indication that the heat treatment had produced a grain size too large to provide a good sample of grains for x-ray examination.

A cross-section of the substrate and deposit was prepared metallographically on another specimen subjected to the same treatment; the photomicrograph after polishing and etching is shown in Figure III-2. This picture shows the chloride deposit, now recrystallized, on top of the still-columnar fluoride substrate. The circular marks are artifacts of the polishing process.

3. Work Function Measurements

The bare work function of the emitter was determined in the converter prior to the introduction of cesium. The calculated results using Richardson's equation are shown in Figure III-3. There was no significant dependence of emitter work function on emitter temperature in the range of 1750 to 1950°K, which indicates the absence of oxygen contamination. The bare work function was found to be about 4.87 eV. After the introduction of cesium, the cesiated work function was determined under highly ion rich conditions and at an interelectrode



spacing of 1 mil. The calculated values of work function are plotted versus the ratio of the emitter to cesium reservoir temperature in Figure III-4.

4. Cesium Families

Families of volt-ampere characteristics were obtained by changing the cesium pressure while the other converter parameters were held constant. Figures III-5 through III-29 show these families in the emitter temperature range of 1600 to 1900°K and the interelectrode spacing range of 0.5 to 40 mils. The emitter temperature indicated represents the temperature at the emitting surface, and the collector temperatures were chosen to be in the vicinity of the optimum values. The output voltage is measured from a voltage tap at the cold end of the emitter sleeve and requires a correction of 1.5 mV per amp/cm² for conversion to electrode voltage. The cesium pressures (P) and the interelectrode spacings (d) in these data are chosen to provide the Pd products of ..., 5, 10, 20, 40, ... mil-torr. A summary of the envelopes of the cesium families is shown in Figures 30 through 33. Each curve in these figures represents the optimized performance with respect to cesium pressure and collector temperature. The fully optimized performance is shown in Figure 34.

5. Spacing Families

Variable-spacing families were obtained covering the spacing range of 0.5 to 40 mils for the cesium pressure range of 0.5 to 4 torr and the ion richness range of 0.1 to 10. Figures 35 through 37 show such families at an ion richness of 10 and cesium pressures of 0.5, 1, and 2 torr. Note that for the Pd of 0.3 mil-torr (Figure 35) there



is a clear saturation and no ignition. At a Pd of 0.6 mil-torr ignition still does not occur, but the saturation current is slightly reduced. The higher Pd curves show a definite ignition, and the observed currents are significantly higher than the saturation current. This is probably caused by ion current and Schottky effects, as suggested by Hansen^{1,2} and Wilkins.³ The spacing families for the ion richness conditions of 1 and 0.1 and the cesium pressure range of 0.5 to 4 torr are shown in Figures 38 through 44. In each family, the extinguished mode and the ignited mode including a large portion of the quasi-saturation region are recorded.

6. Summary of the Data

Based on the work function measurements, an emission map of the surface was constructed, as shown in Figure 45. The electron and ion currents are shown by the dashed and the solid curves, and the ion richness is indicated by the solid lines. Emitter work function (the open circles) measurements were performed under highly rich conditions and cesium families (the open triangles) were obtained mostly under electron rich conditions. A total of 10 spacing families (solid squares) were obtained, 3 at the ion richness of 10, 4 at the ion richness of 1, and 3 at the ion richness of 0.1. This set of data provides a large number of volt-ampere characteristics at the various combinations of Pd, P and β , and is useful for theoretical analyses and correlations.

7. Correlations

Several years ago, Kitrilakis⁴ found a correlation between the deviation from ideal performance (V^*) and the product of cesium



pressure and interelectrode spacing (Pd). He stated, however, that the correlation was only true within a band of 0.1 volt. Using the present set of data, an attempt was made to determine if there are other dependencies contributing to the band. For this purpose, the voltage loss V^* was defined as the difference in voltage between the Boltzmann curve and the J-V curve at the point of tangency to the cesium optimized envelope (Figure 46). According to this definition, V^* is a measure of the deviation from ideal performance, and hence it is useful for indicating trends that may lead to an improvement in performance. The Boltzmann curve was established by the equation:

$$J = AT_e^2 \exp \left(\frac{\phi_c + V}{kT_e} \right)$$

where J is electron current, A is Richardson constant, T_e is emitter temperature, k is Boltzmann constant, V is electrode voltage, and ϕ_c is the collector work function for molybdenum.⁵ Note that a correction of 1.5 mV per amp/cm² must be added to the output voltage of the J-V curves to obtain electrode voltage.

A summary of the results at the ion richness of 0.01 and the cesium pressures of 2, 4, and 8 is presented in Figure 47. The curves with lower cesium pressures exhibit a lower V^* , but since each pressure corresponds to a particular emitter temperature (Figure 47), the question arises whether this behavior is due to cesium pressure or emitter temperature. Similar plots made at constant emitter temperatures also showed a decrease in V^* with decreasing P; hence this effect appears to be mainly due to cesium pressure. The data for the ion richness conditions of 0.05, 0.1 and 0.5 are shown in Figures 48 through 50. Note that at the ion richness of 0.5 the dependence of V^*



on P begins to break down, probably because of approaching neutral conditions. A cross plot of the data in Figure 51 indicates that at constant values of Pd, the voltage loss is an increasing function of cesium pressure. It also appears that β in the range of 10^{-2} to 10^{-1} has little influence on V^* .

B. VACUUM WORK FUNCTION MEASUREMENT APPARATUS

The apparatus described in the Second Quarterly Report on this program has been debugged and put into operation (Figure III-52). It has demonstrated the problems usually associated with new high-temperature, high-vacuum equipment, which have gradually been eliminated, and it is now providing useful data. Emitter temperatures up to 2500°K can be obtained easily. The system pressure depends chiefly upon the length of time it has been operating, and reached 10^{-7} torr at operating temperatures overnight. Pressures a decade lower can be obtained with patience.

Temperature measurements have been made through a side-window using a micro-pyrometer sighted on a radial blackbody cavity (L/D at least 8:1). Measurements are made by using a variable power supply to obtain an I-V curve for the device. The current signal is obtained from an appropriate resistor (1, 10, or 100Ω) in the circuit, and the guard is maintained at collector potential except for the drop through the resistor, which is always less than 50 mV (see Figure III-53).

Preliminary data were obtained from a specimen of ground and annealed tungsten vapor-deposited from the fluoride. The surface work function is obtained from Richardson's equation and the measured values of saturation current and surface temperature. A summary of the



data is shown in Figure III-54. The diagonal lines represent constant values of work function. At emitter temperatures higher than 1850°K, the data points follow the work function line of 4.51 within ± 0.01 eV, indicating that the partial pressure of oxygen at these temperatures is sufficiently low and does not interfere with the bare work function measurements. For emitter temperatures lower than 1850°K, the data points begin to drift towards higher work function lines, since the surface temperature is low and oxygen begins to adhere to the surface. Work function measurements on similar emitters, performed at Gulf General Atomics, have shown a work function range of 4.53 to 4.50 eV.

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1. Hansen, L. K., "Anomalous Shottky Effect," Journal of Applied Physics, Vol. 37, No. 12, November 1966, pp. 4498 - 4502.
2. Hansen, L. K., "Ion Current Effects in Cesium Diodes," Report on 25th Annual Conference on Physical Electronics, MIT, March 1965.
3. Wilkins, D. R., "An Improved Theoretical Description of Thermionic Converter Performance Characteristics," Report on 27th Annual Conference on Phys. Electronics, MIT, March 1967.
4. S. S. Kitrilakis and J. H. Weinstein, Second Annual Technical Summary Report for the Thermionic Emitter Materials Research Program, No. TE27-64, to the Office of Naval Research, September 1963.
5. A. Shavit and S. S. Kitrilakis, Report on the Effect of Electrode Configuration on Thermionic Converter Output Characteristics, No. TE7-65, to the Air Force Cambridge Research Laboratories, March 1964.

Acknowledgment

The authors would like to thank A. Donaker for assistance in taking and reducing the data.

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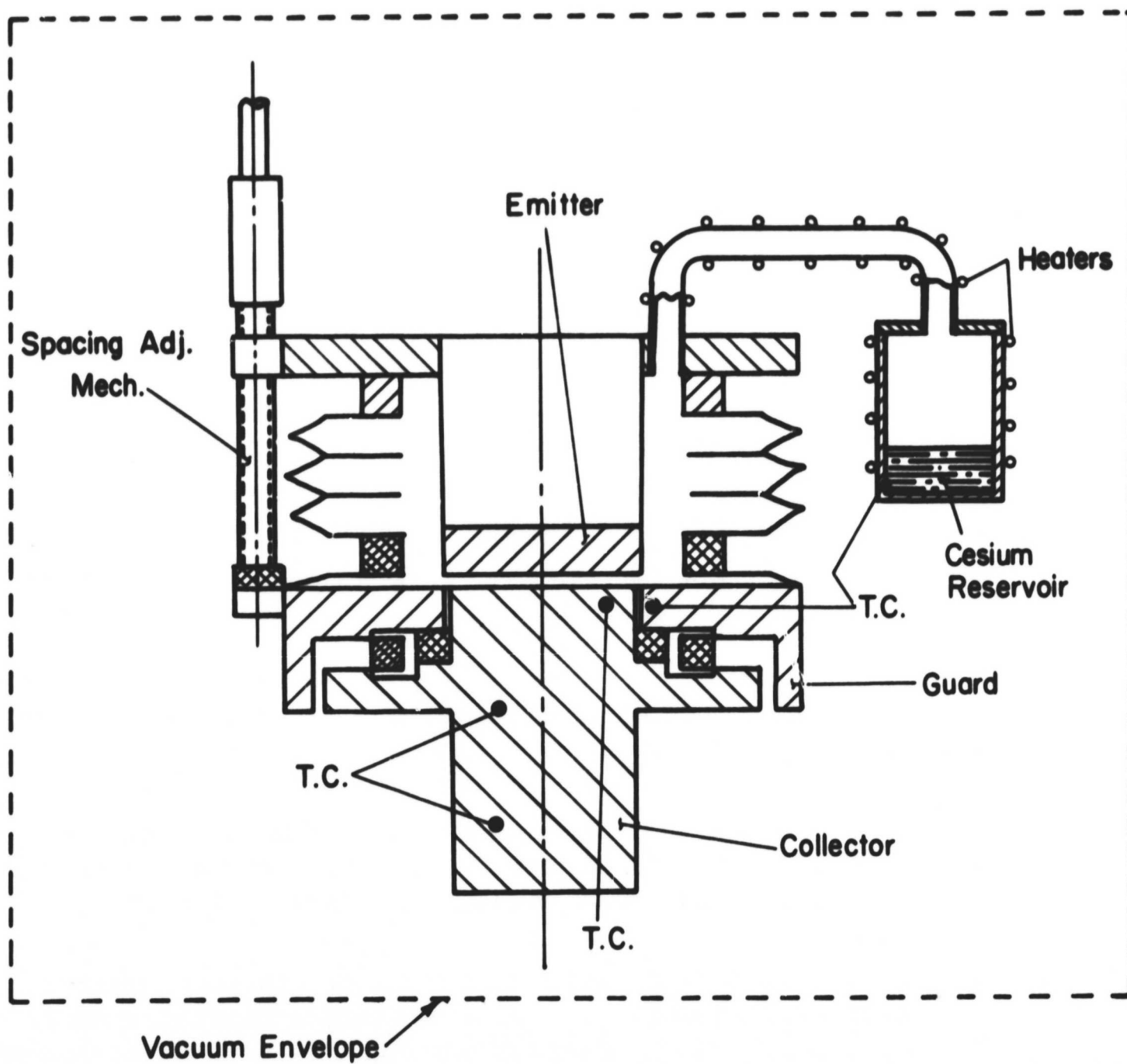


Figure III-1 Schematic Diagram of a Research Thermionic Converter.

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**Figure III-2 Cross-Section of Layer Vapor-Deposited
from the Chloride to Make Surface of Emitter.**

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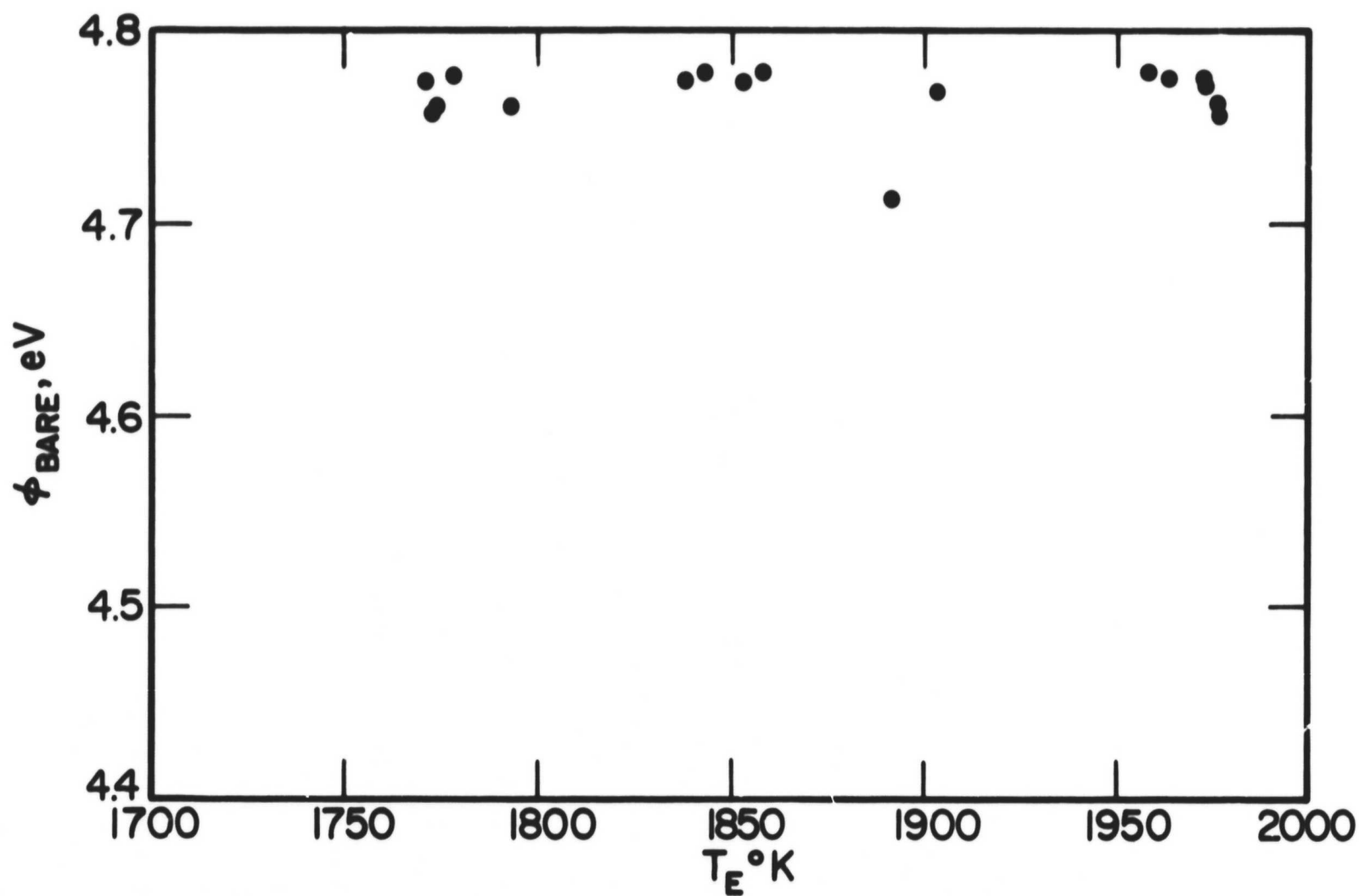


Figure III-3 Summary of Bare Work Function Data.

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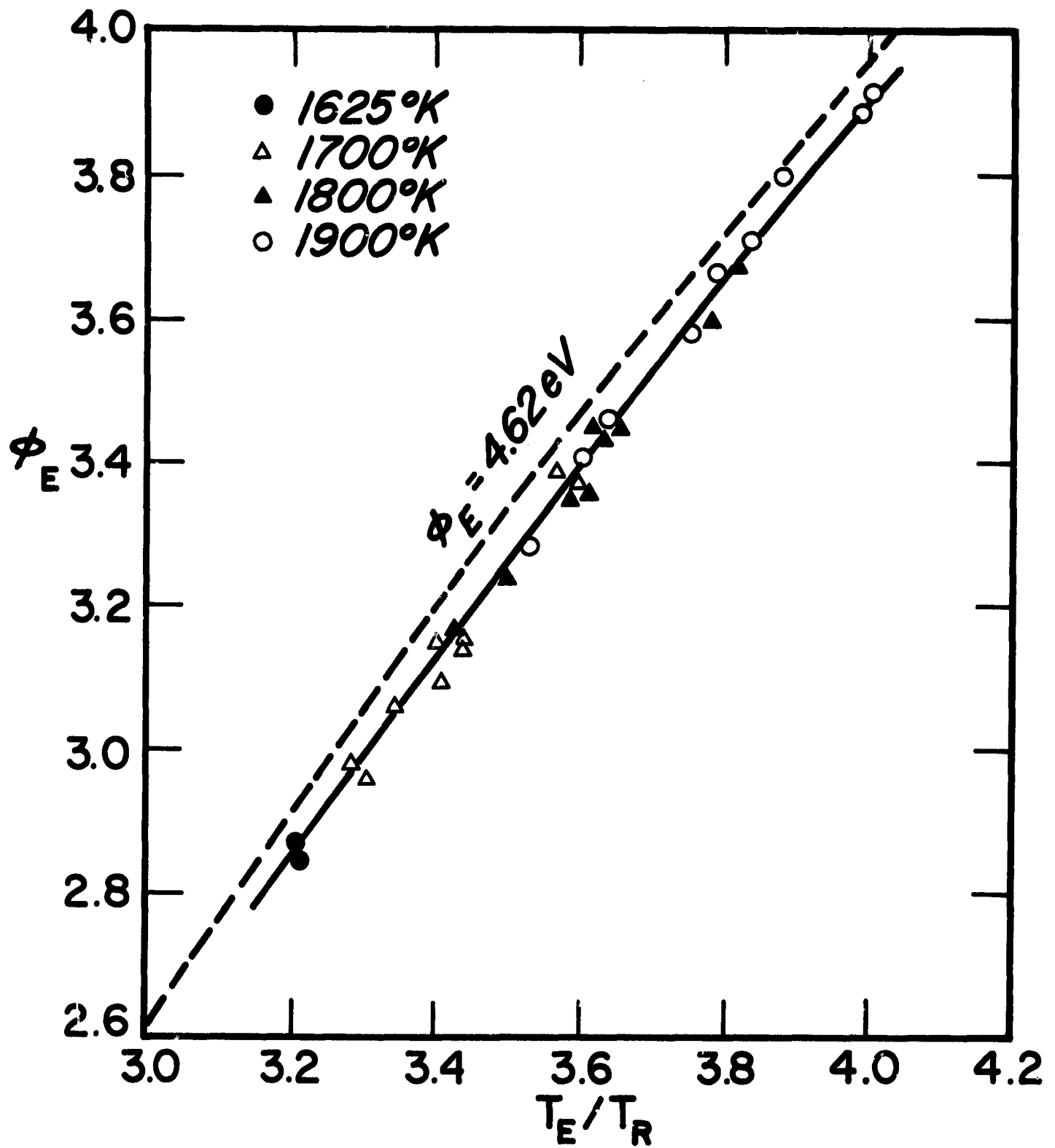


Figure III-4. Emitter Work Function Data.

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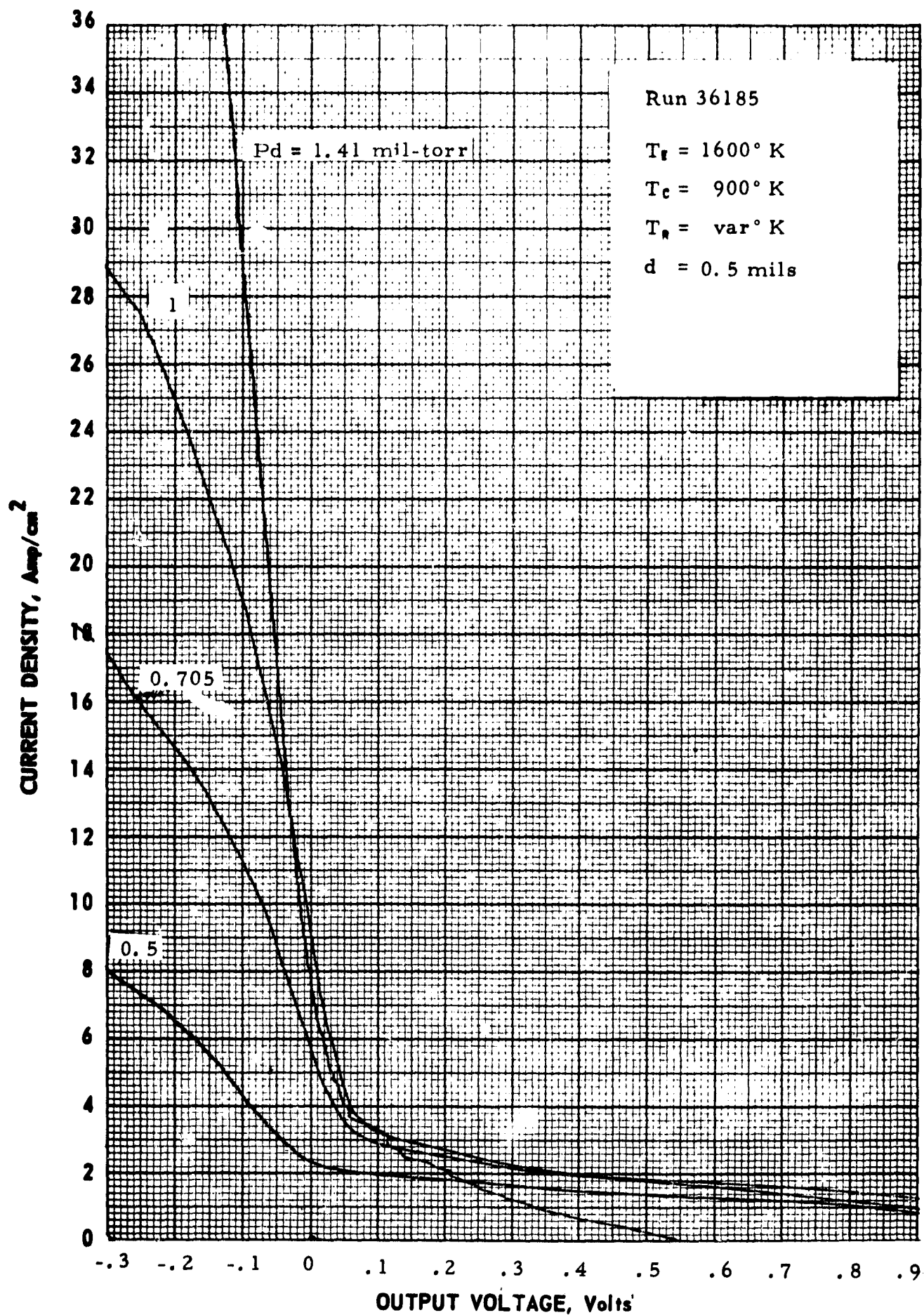


Figure III-5. Variable Cesium Pressure Family at T_f = 1600° K and d = 0.5 mils.

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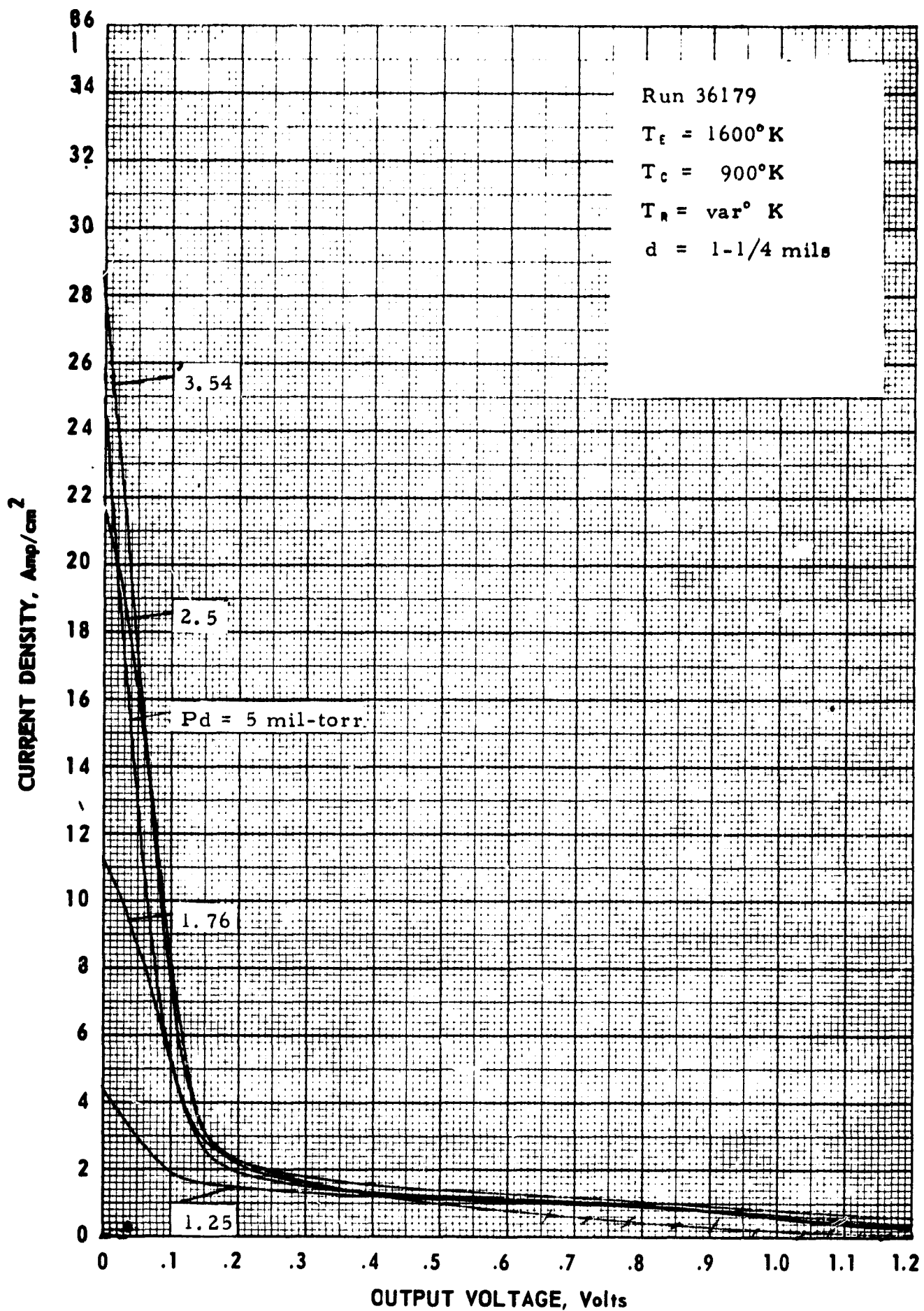


Figure III-6. Variable Cesium Pressure Family at $T_f = 1600^\circ\text{K}$ and $d = 1.25 \text{ mils}$.



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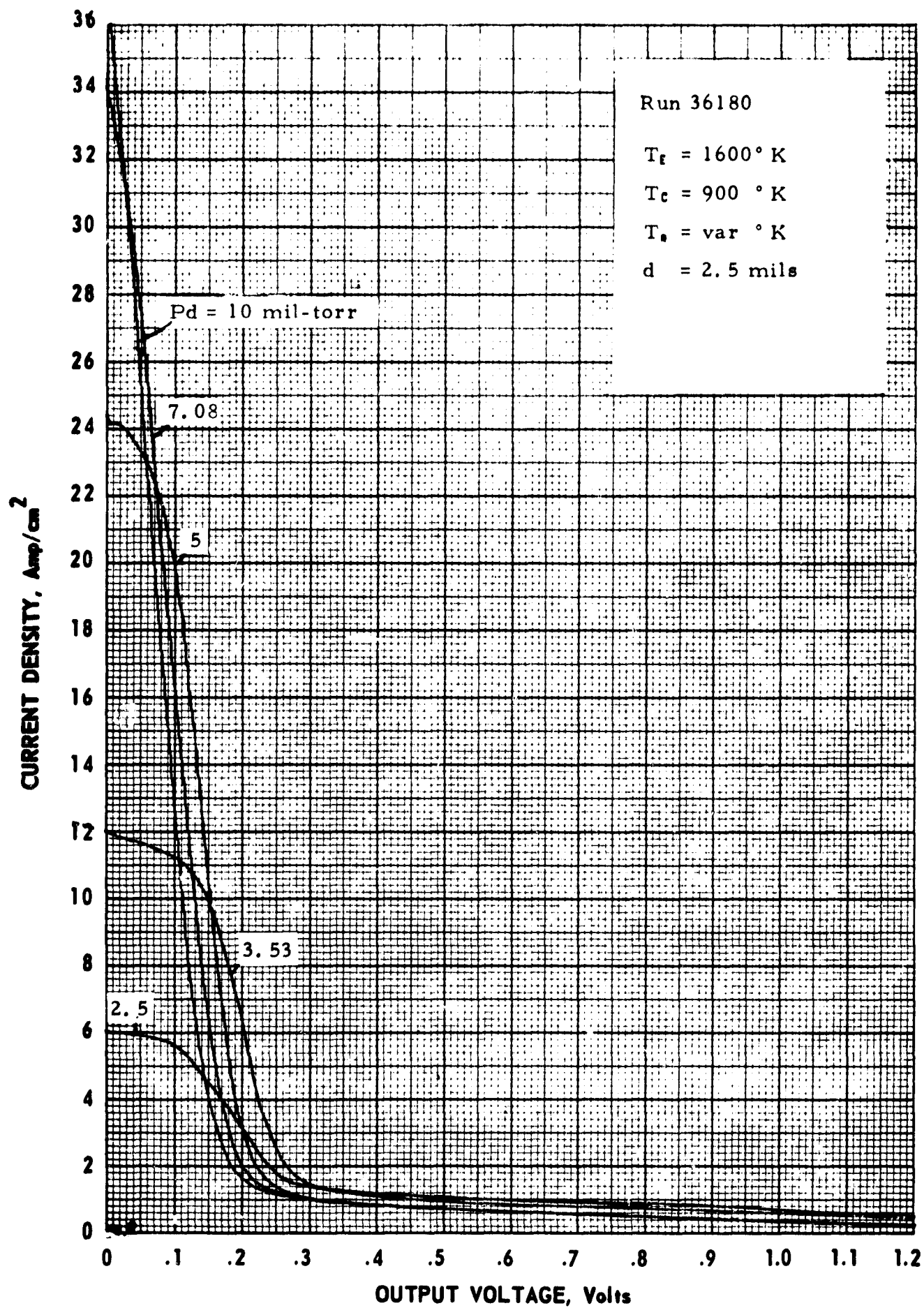


Figure III-7. Variable Cesium Pressure Family at $T_f = 1600^\circ \text{K}$ and $d = 2.5 \text{ mils}$.

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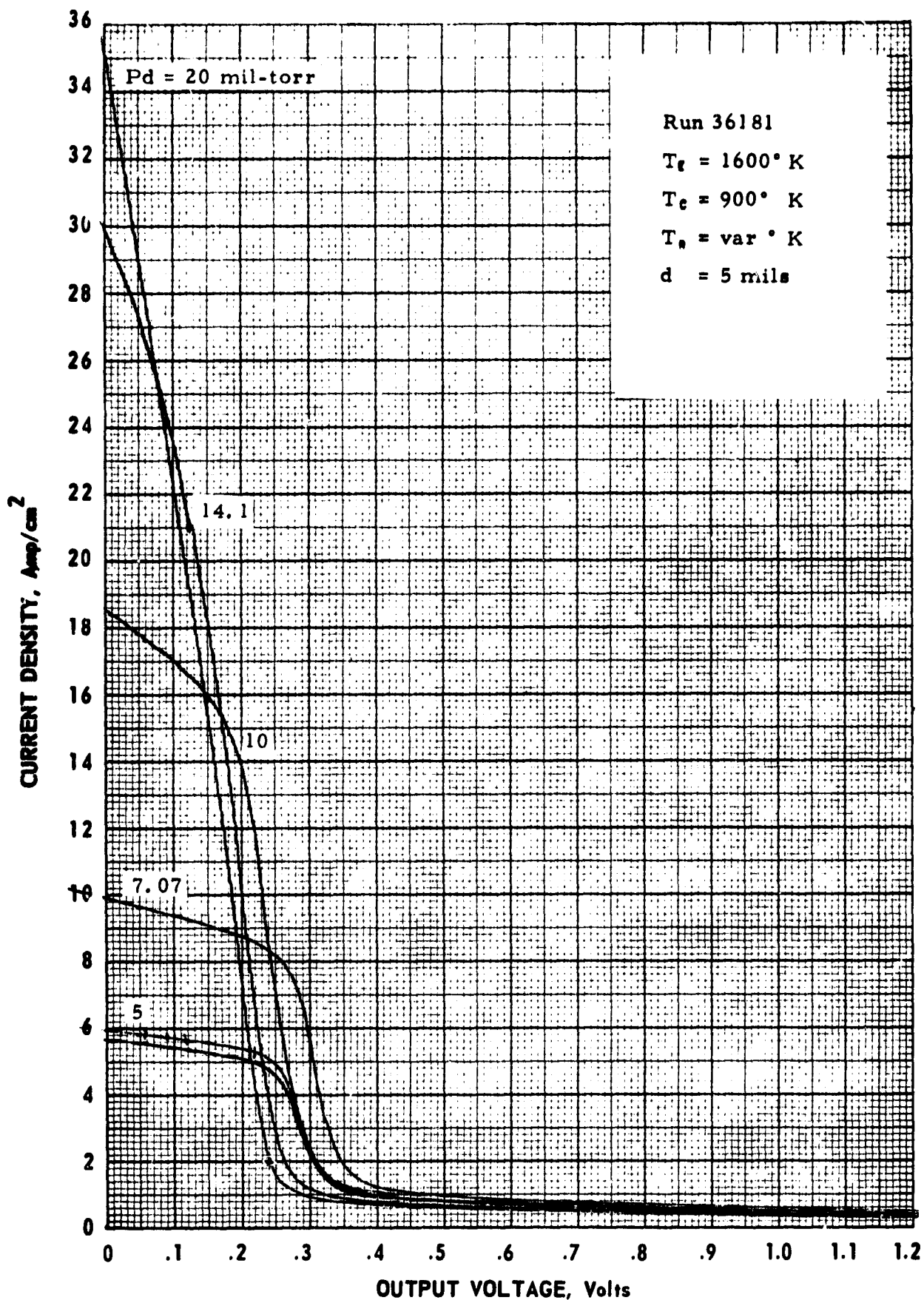


Figure III-8. Variable Cesium Pressure Family at $T_c = 1600^\circ \text{ K}$ and $d = 5 \text{ mils}$.

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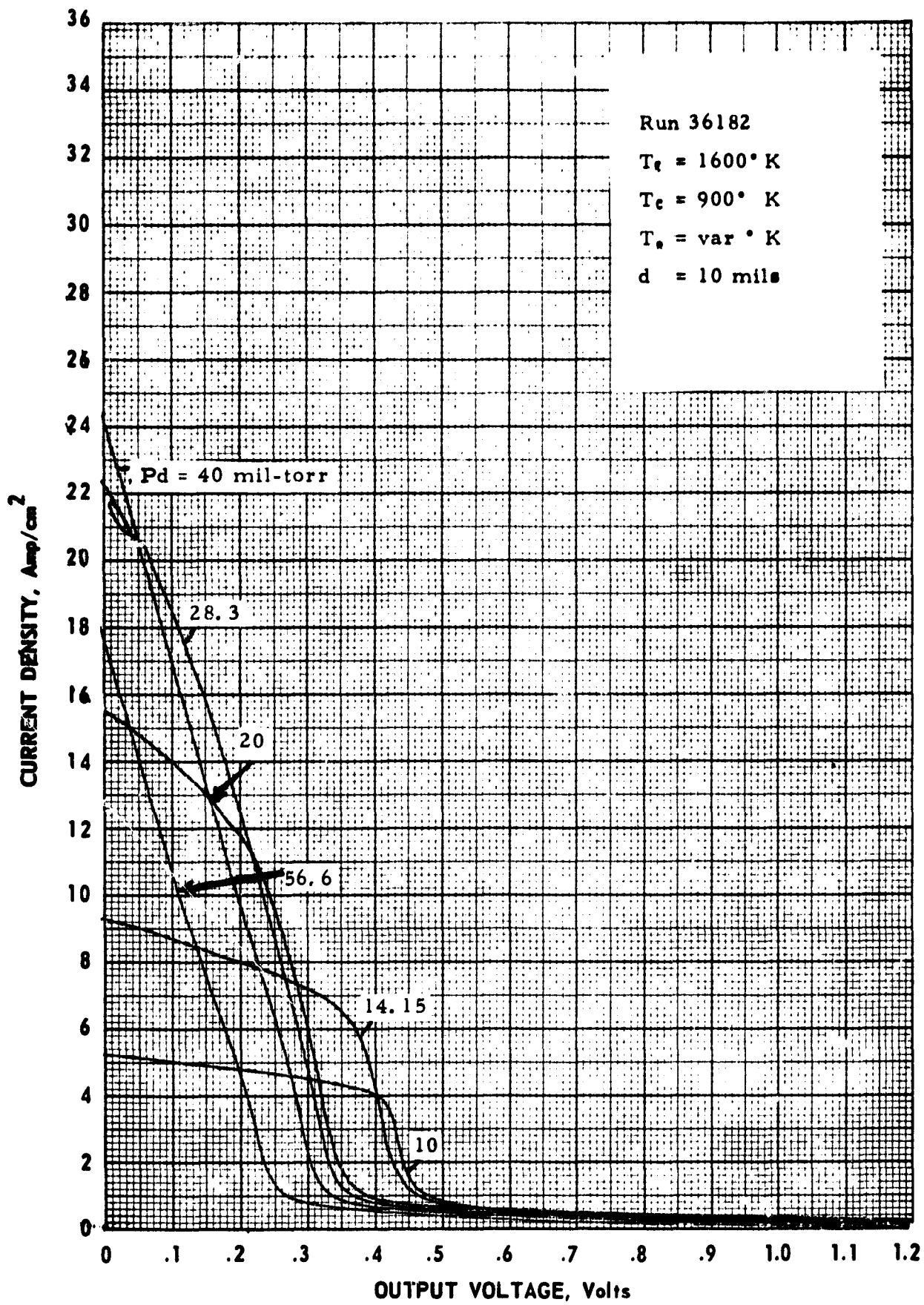


Figure III-9. Variable Cesium Pressure Family at $T_g = 1600^\circ \text{ K}$ and $d = 10 \text{ mils}$.



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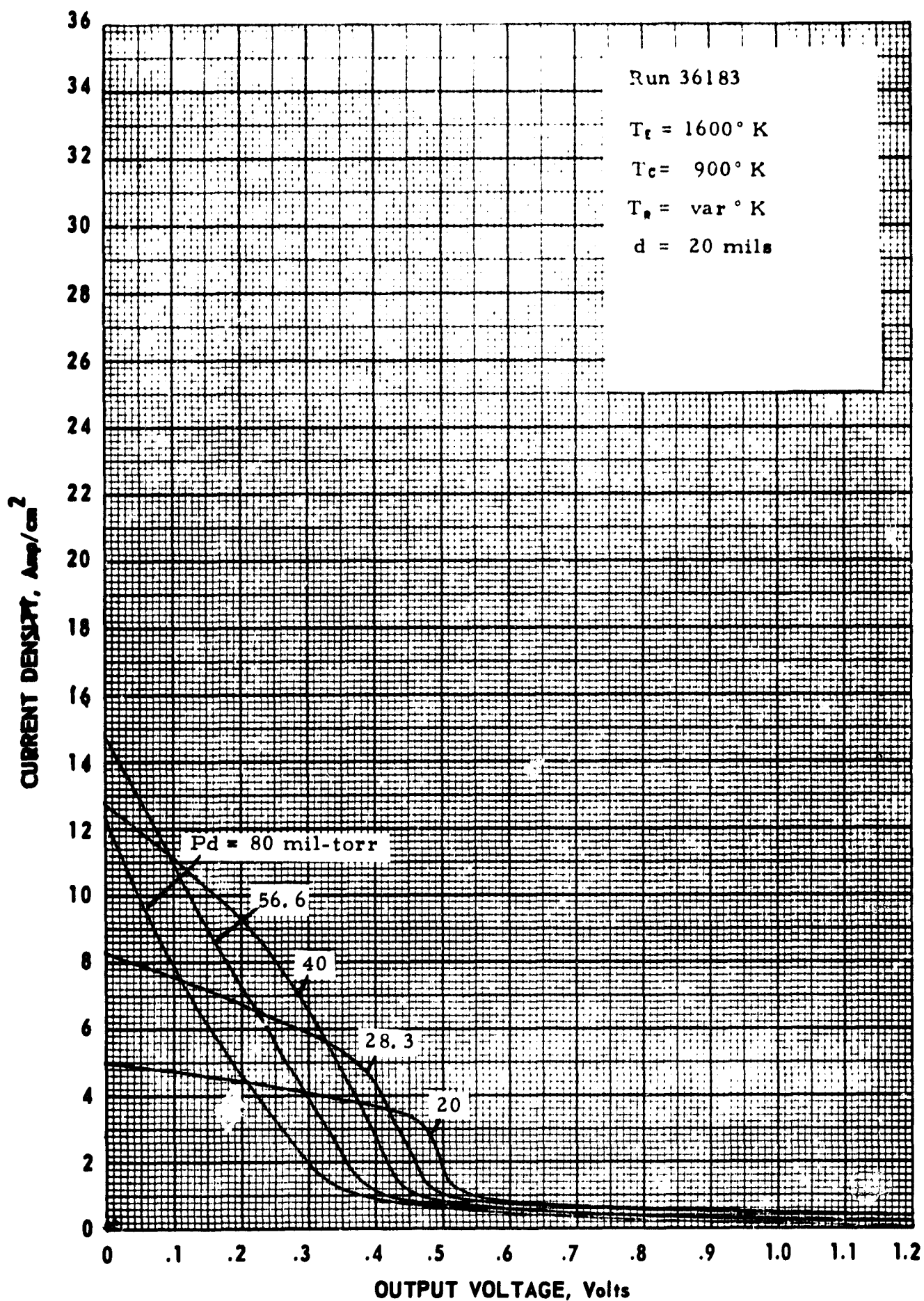


Figure III-10. Variable Cesium Pressure Family at $T_f = 1600^\circ \text{K}$ and $d = 20 \text{ mils}$.

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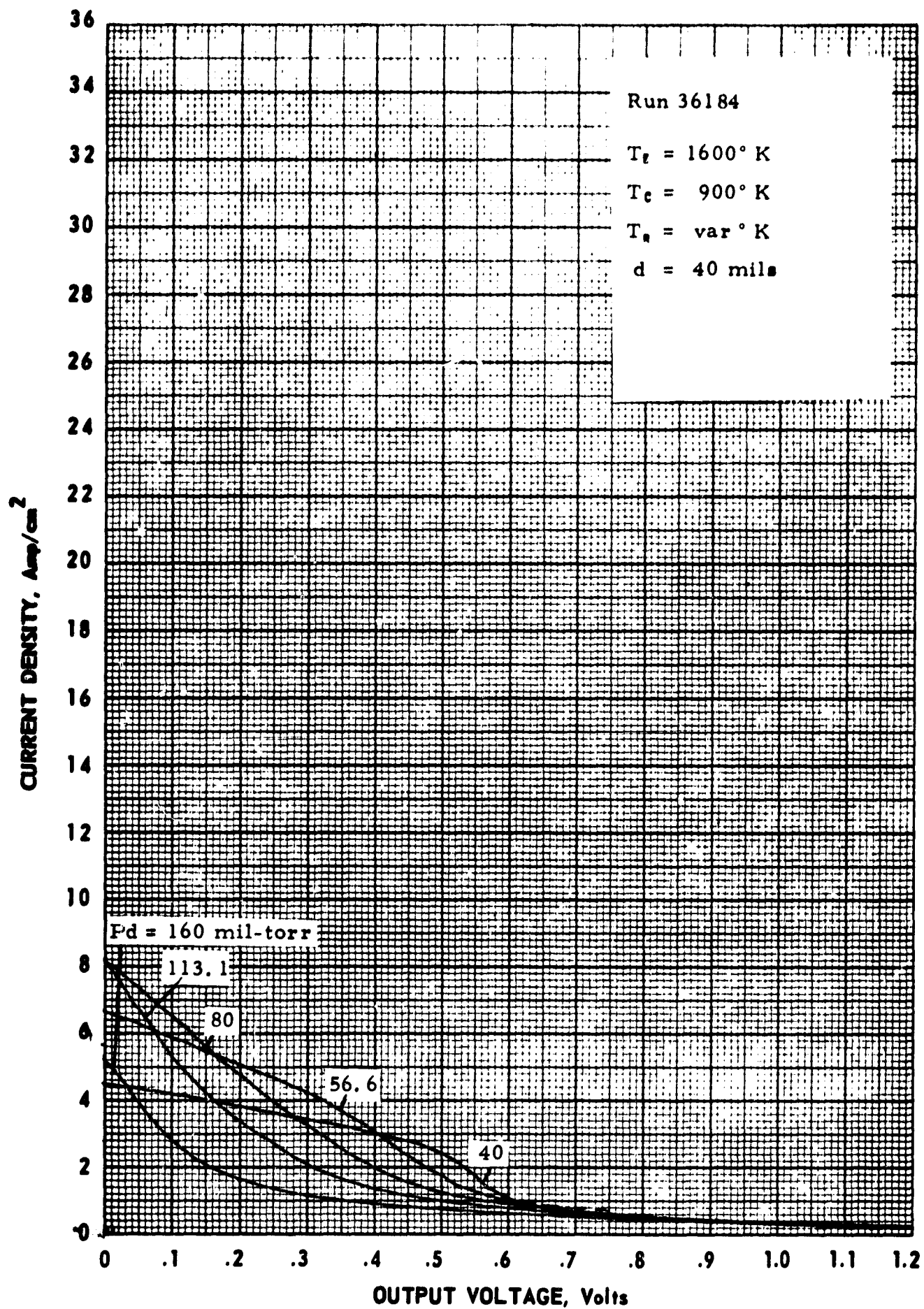


Figure III-11. Variable Cesium Pressure Family at $T_f = 1600^\circ \text{K}$ and $d = 40 \text{ mils}$.



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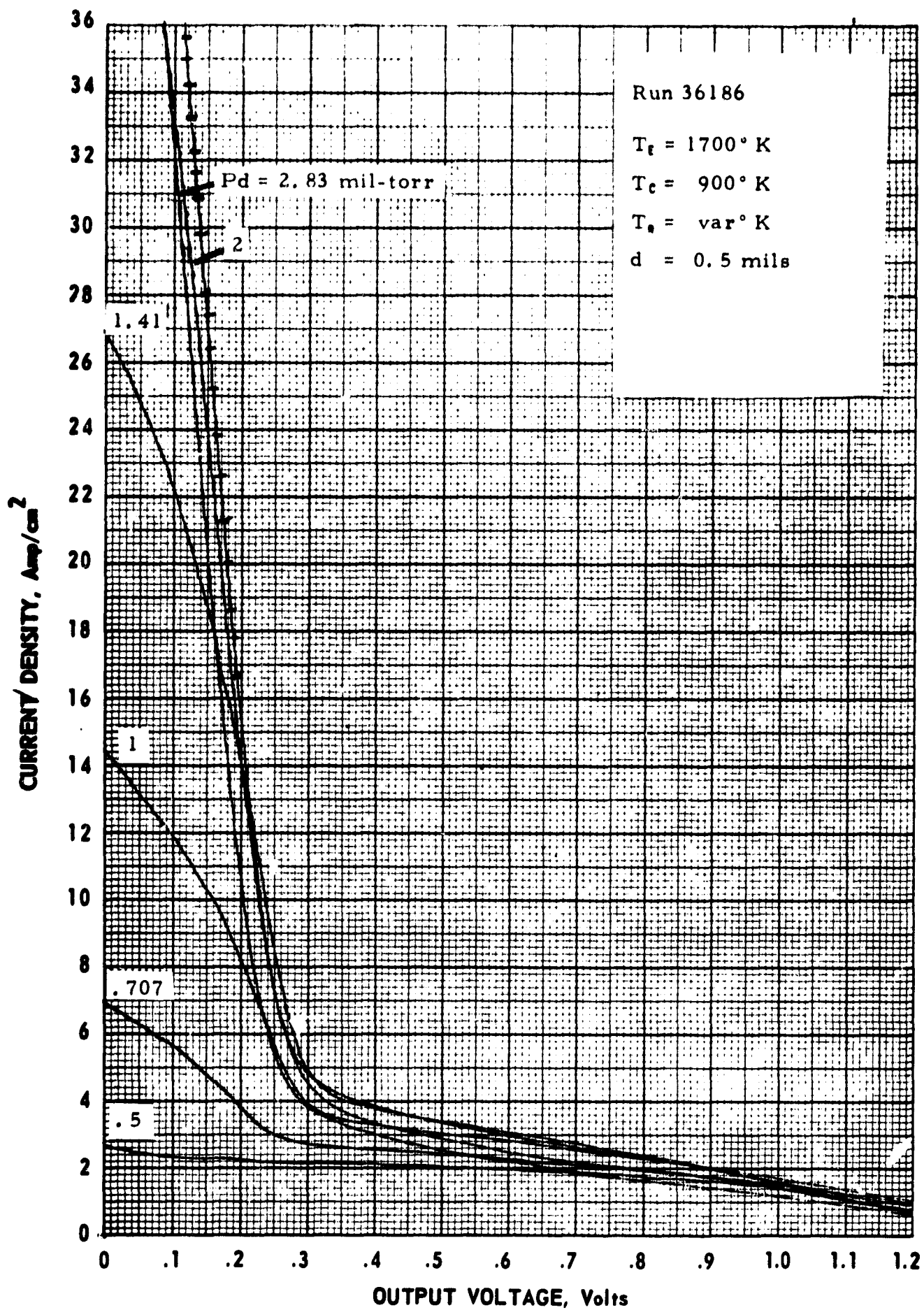


Figure III-12. Variable Cesium Pressure Family at $T_f = 1700^\circ \text{K}$ and $d = 0.5 \text{ mils}$.



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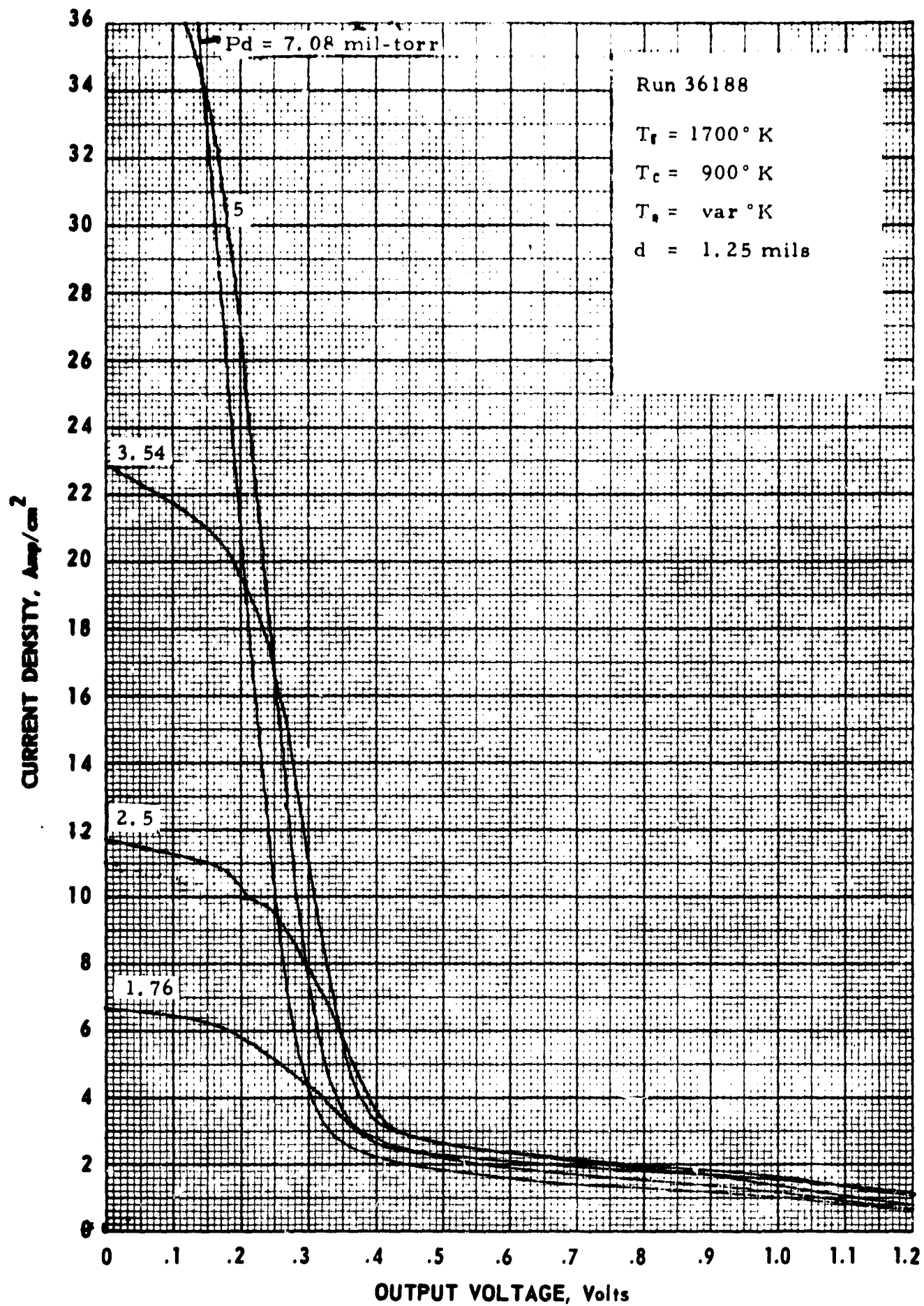


Figure III-13. Variable Cesium Pressure Family at $T_f = 1700^\circ \text{K}$ and $d = 1.25 \text{ mils}$.

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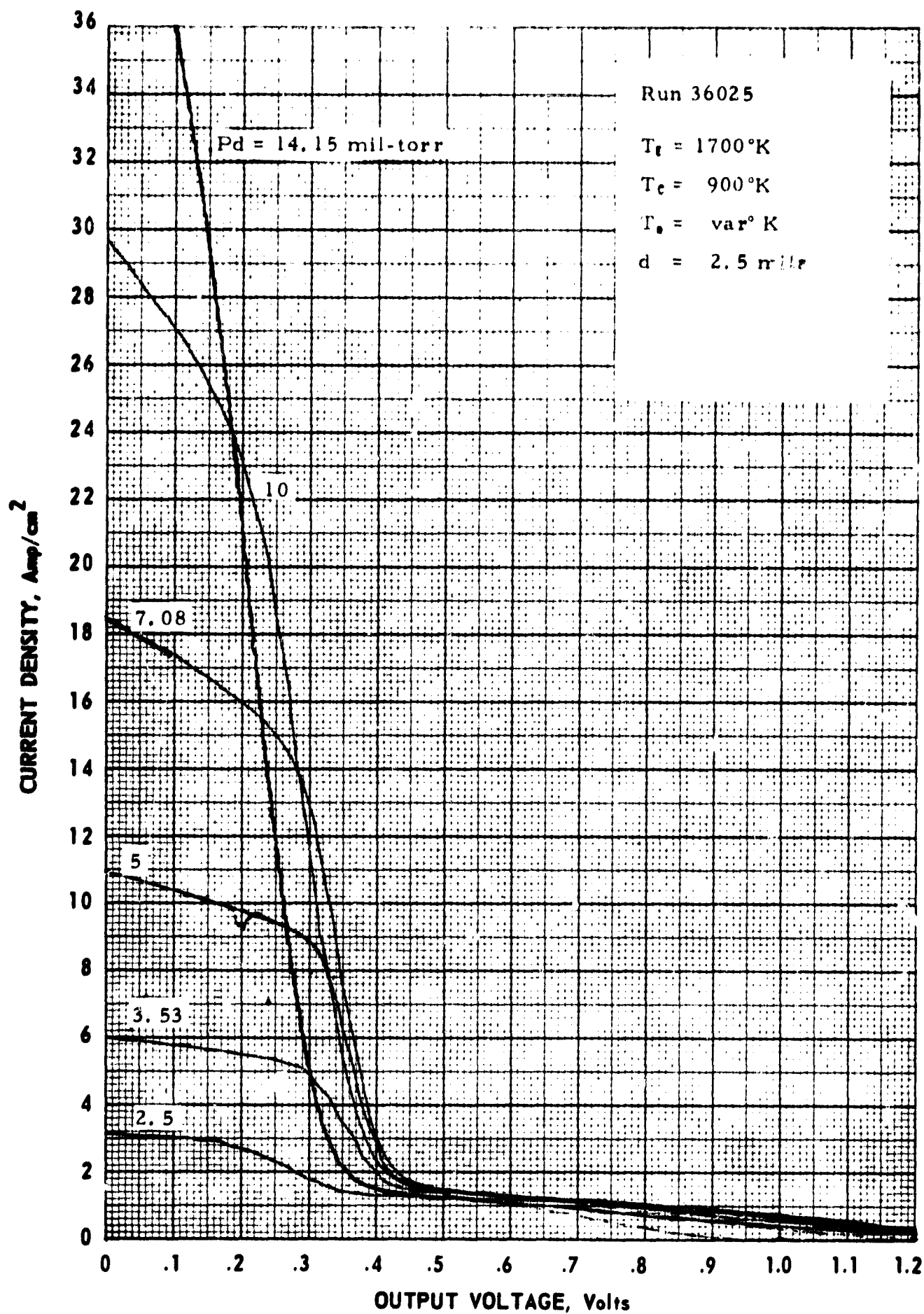


Figure III-14. Variable Cesium Pressure Family at $T_e = 1700^\circ\text{K}$ and $d = 2.5 \text{ mils}$.

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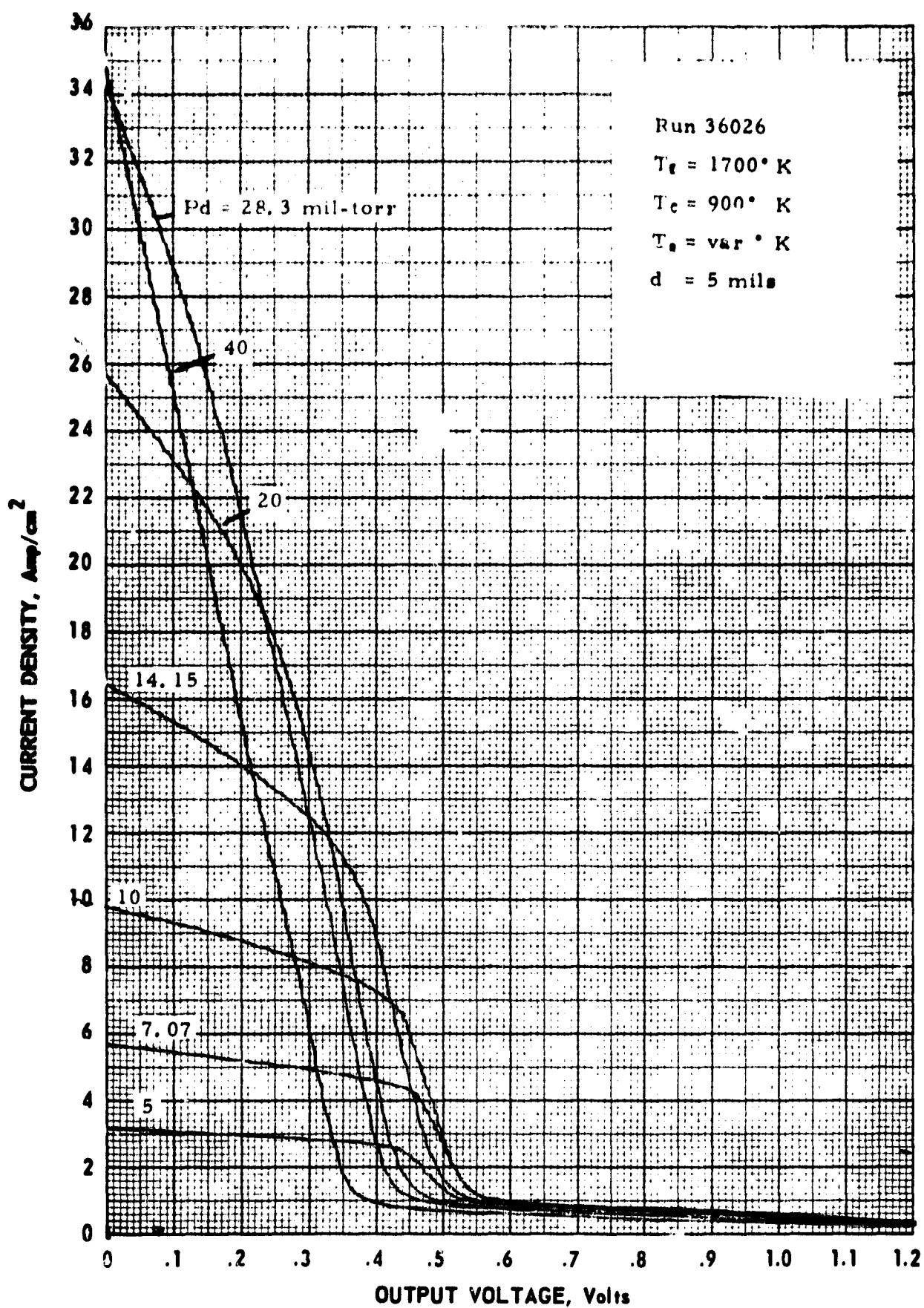


Figure III-15. Variable Cesium Pressure Family at $T_f = 1700^\circ \text{K}$ and $d = 5 \text{ mils}$.

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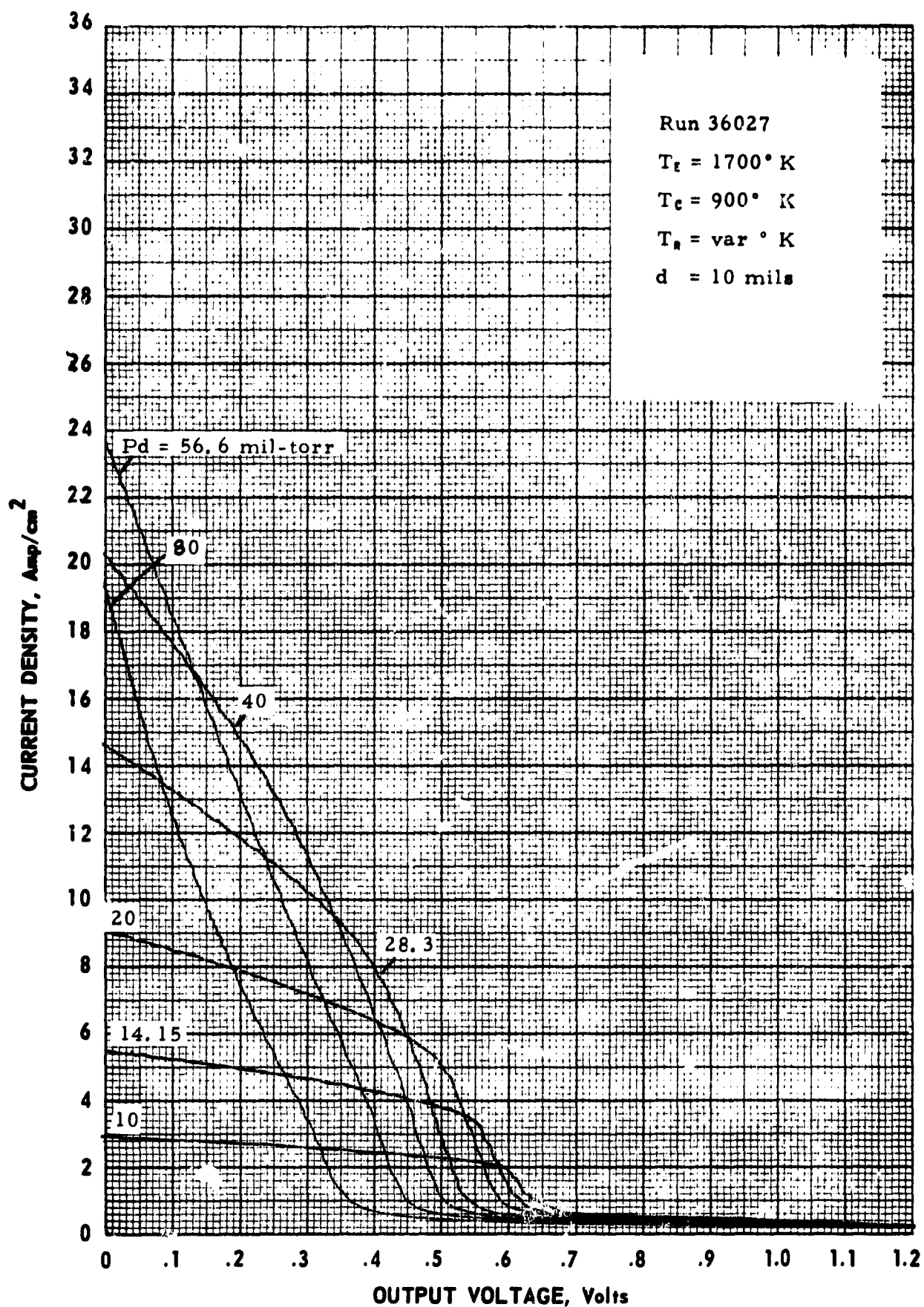


Figure III-16. Variable Cesium Pressure Family at $T_f = 1700^\circ \text{ K}$ and $d = 10 \text{ mils}$.



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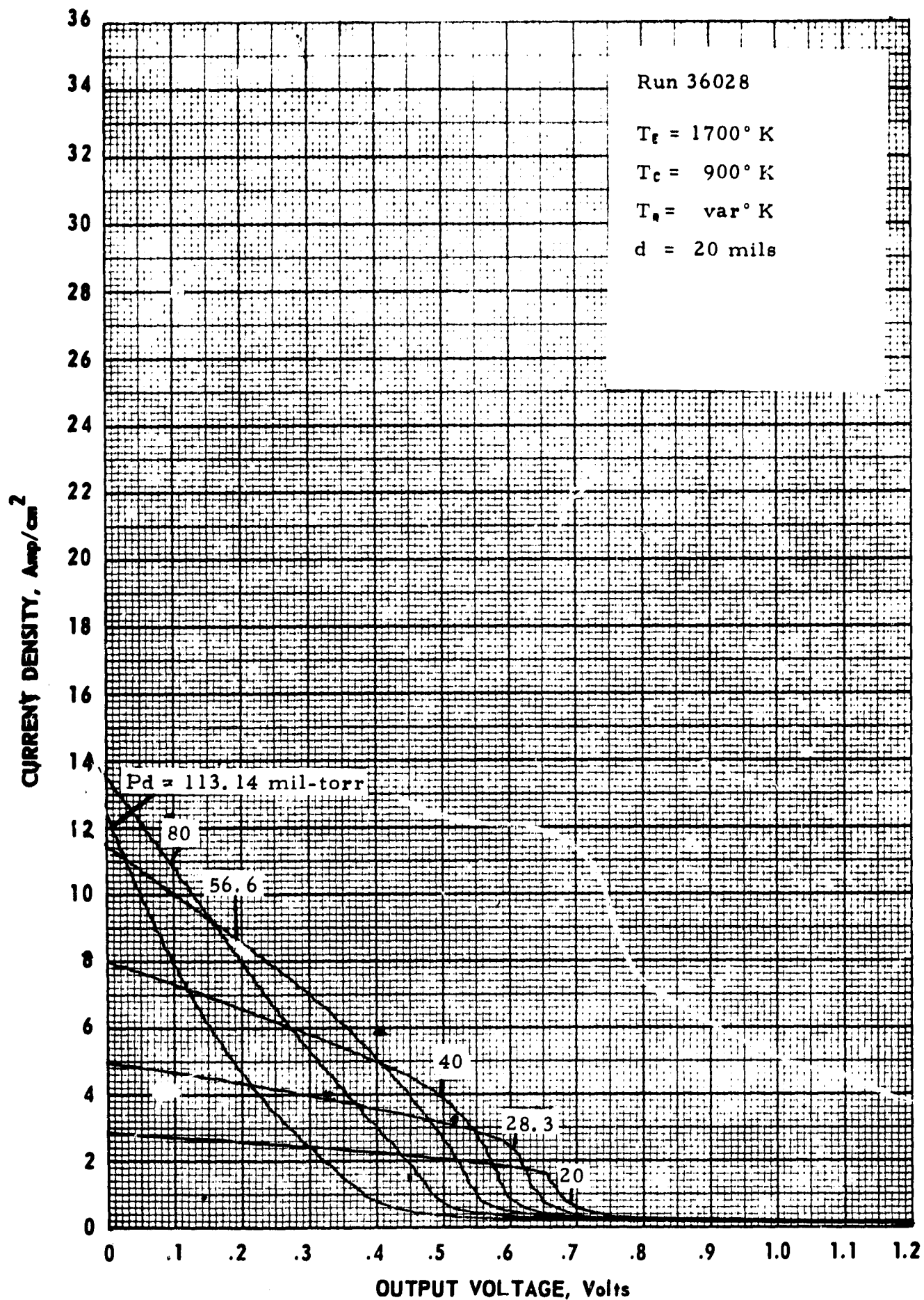


Figure III-17. Variable Cesium Pressure Family at $T_f = 1700^\circ \text{K}$ and $d = 20 \text{ mils}$.

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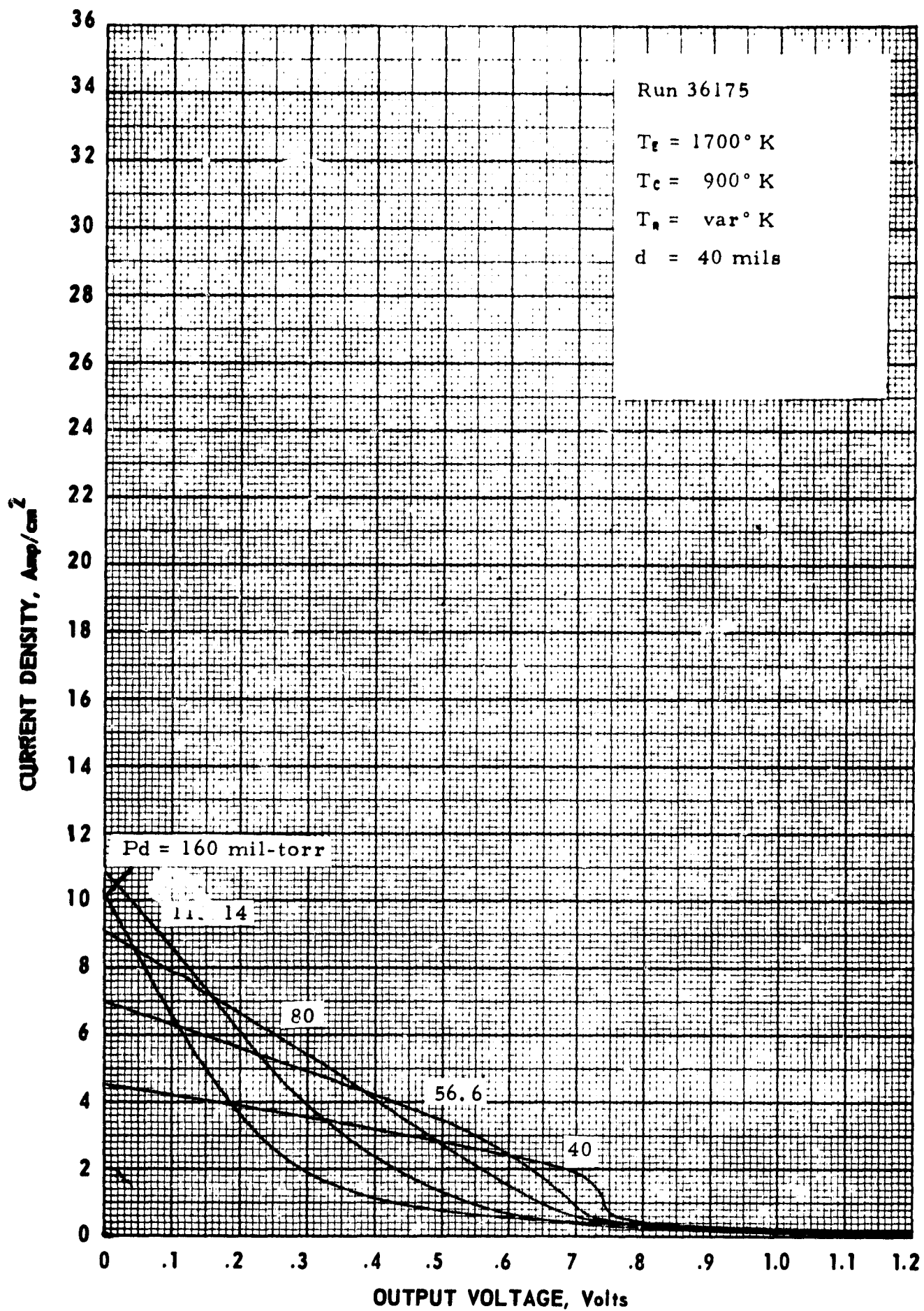


Figure III-18. Variable Cesium Pressure Family at $T_e = 1700^\circ \text{ K}$ and $d = 40 \text{ mils}$.

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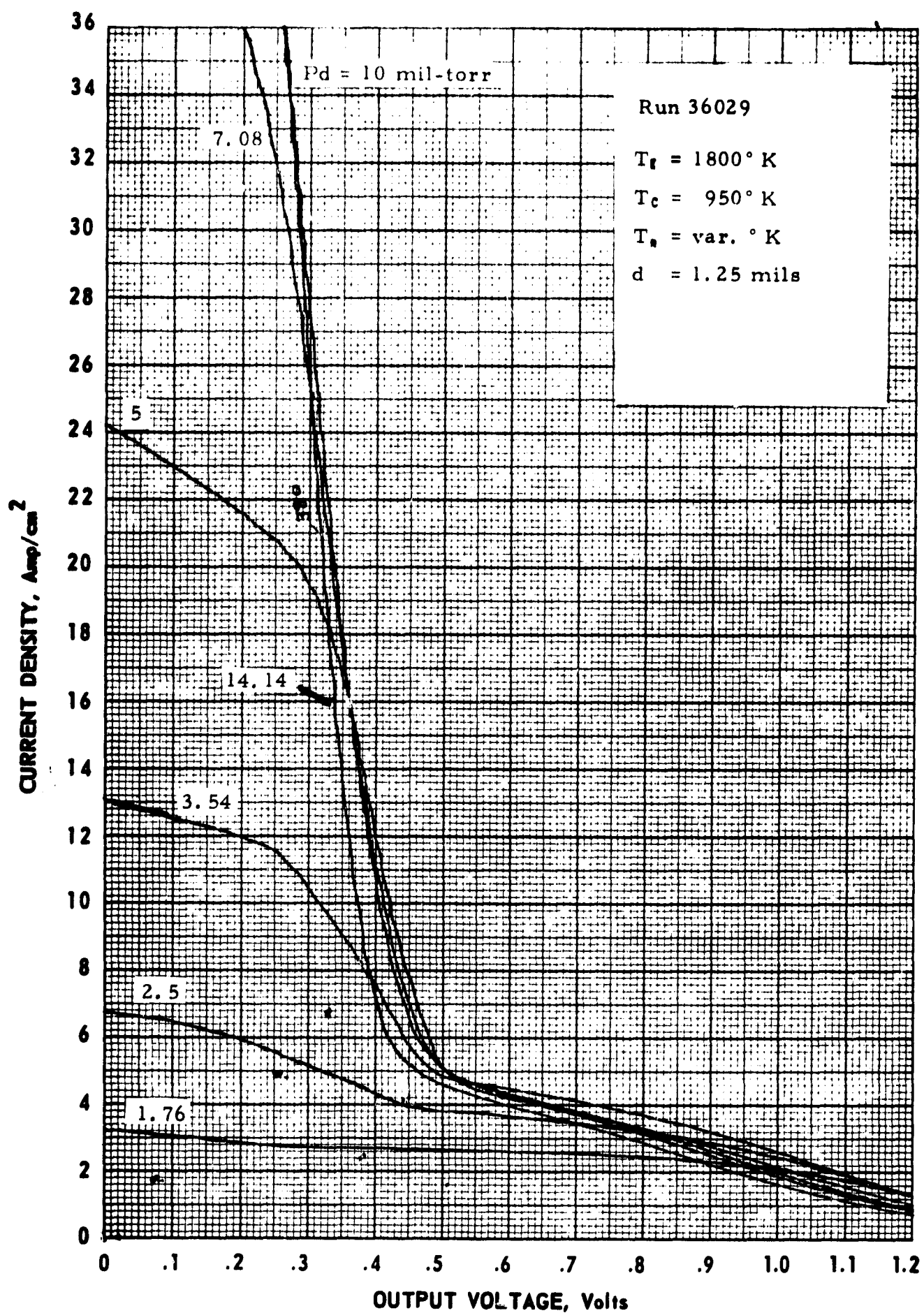


Figure III-19. Variable Cesium Pressure Family at $T_f = 1800^\circ \text{K}$ and $d = 1.25 \text{ mils}$.



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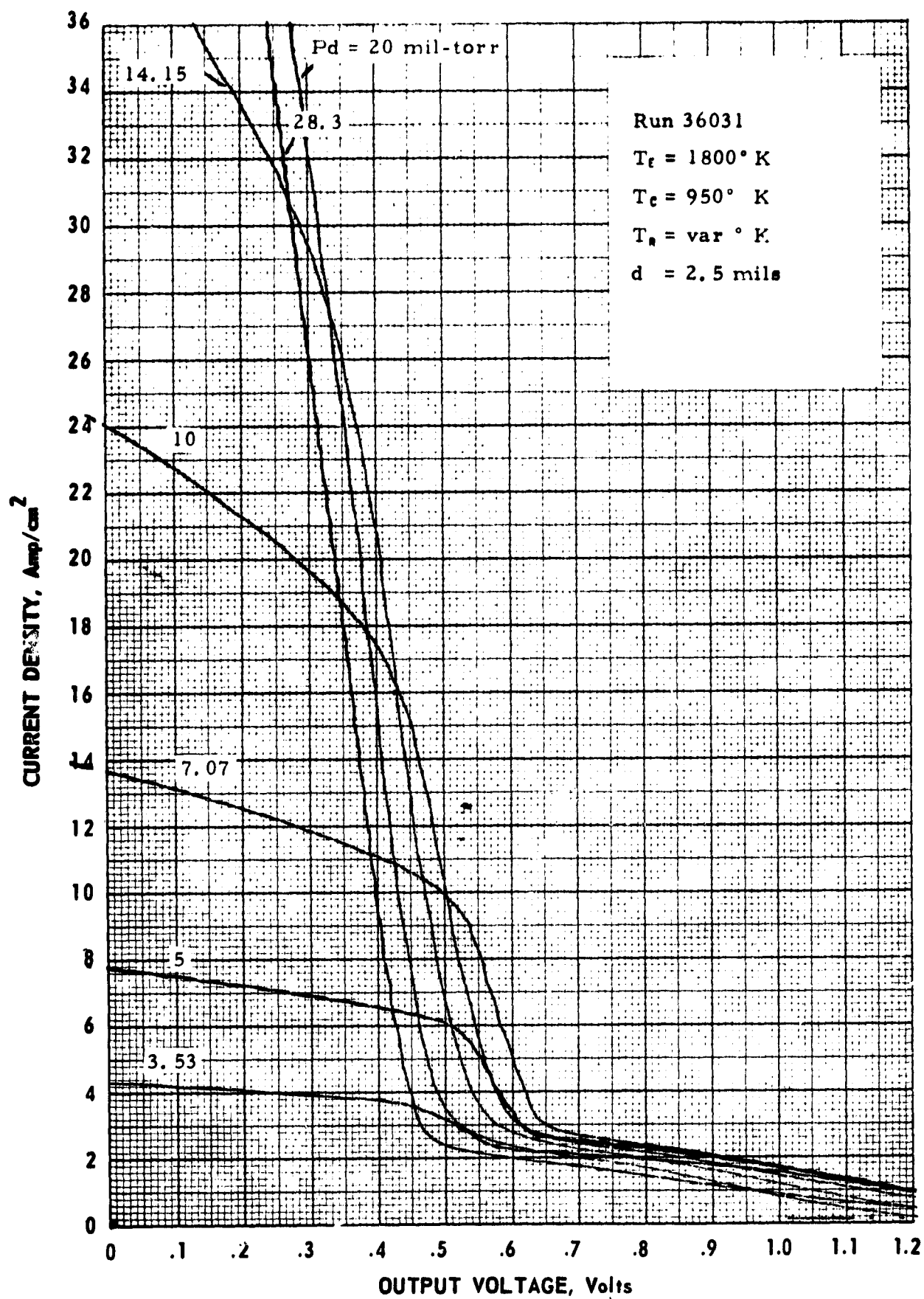


Figure III-20, Variable Cesium Pressure Family at $T_e = 1800^\circ \text{K}$ and $d = 2.5 \text{ mils}$.



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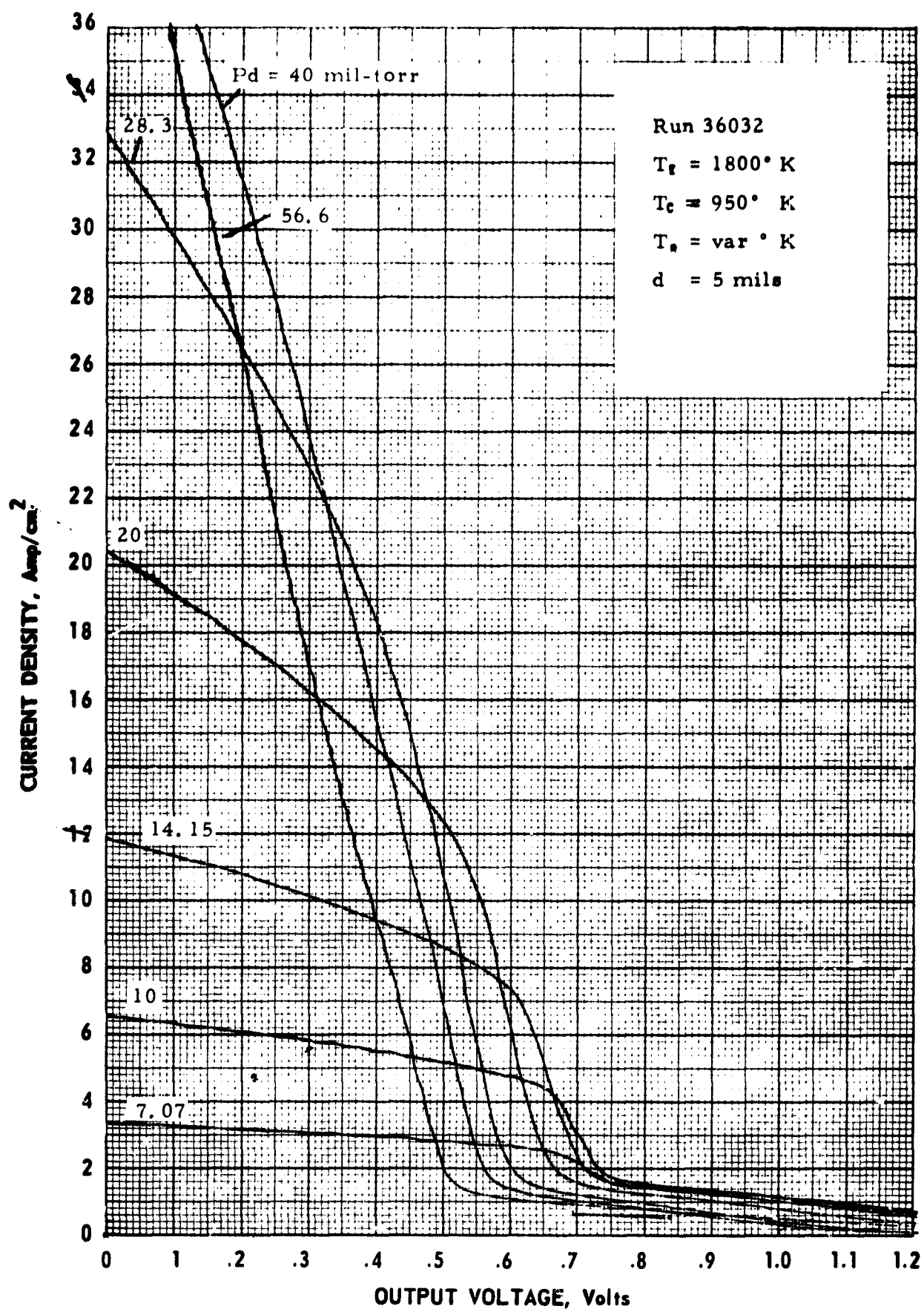


Figure III-21. Variable Cesium Pressure Family at $T_e = 1800^\circ \text{ K}$ and $d = 5 \text{ mils}$.

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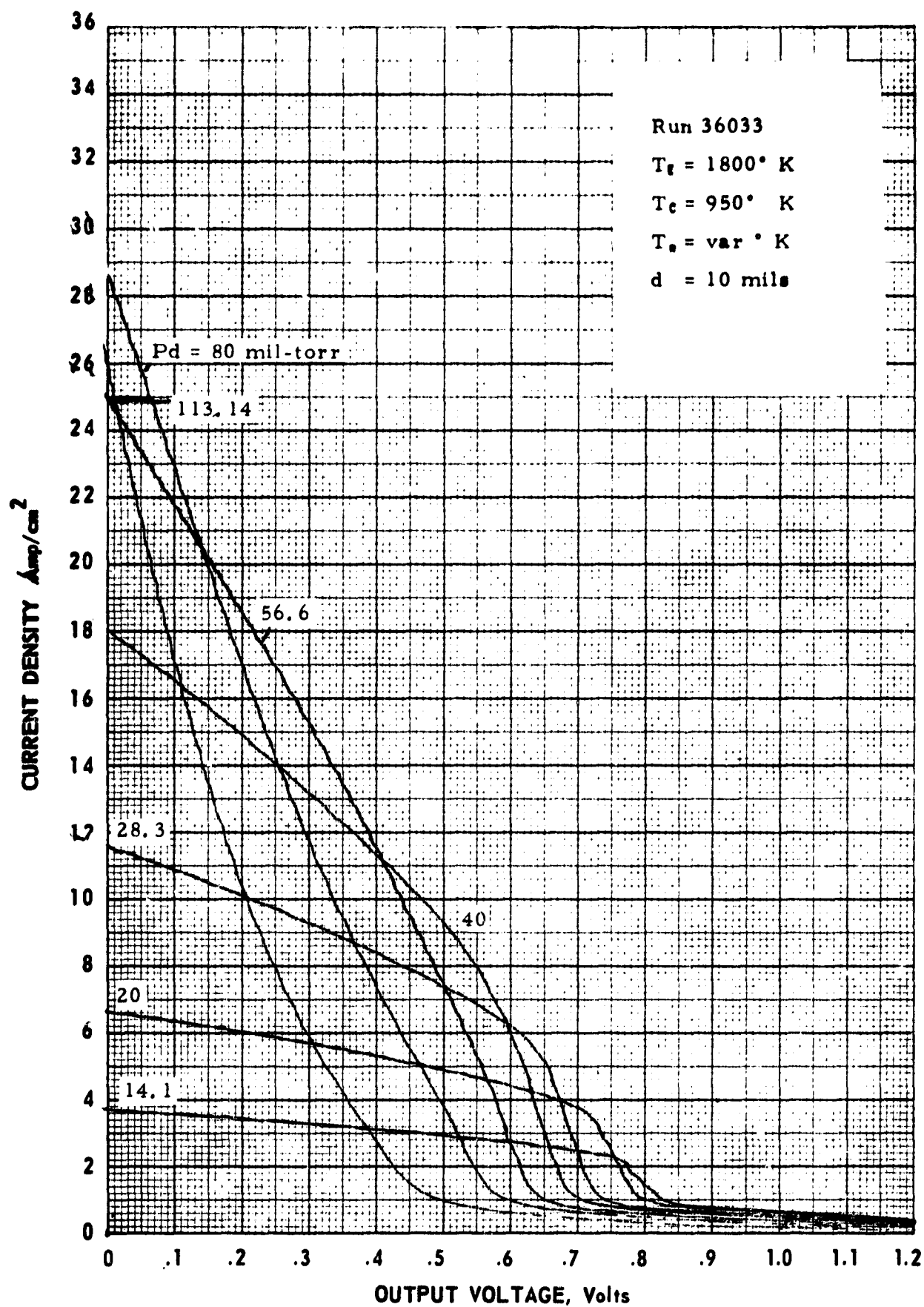


Figure III-22. Variable Cesium Pressure Family at $T_e = 1800^\circ \text{ K}$ and $d = 10 \text{ mils}$.

68-TR-11-10

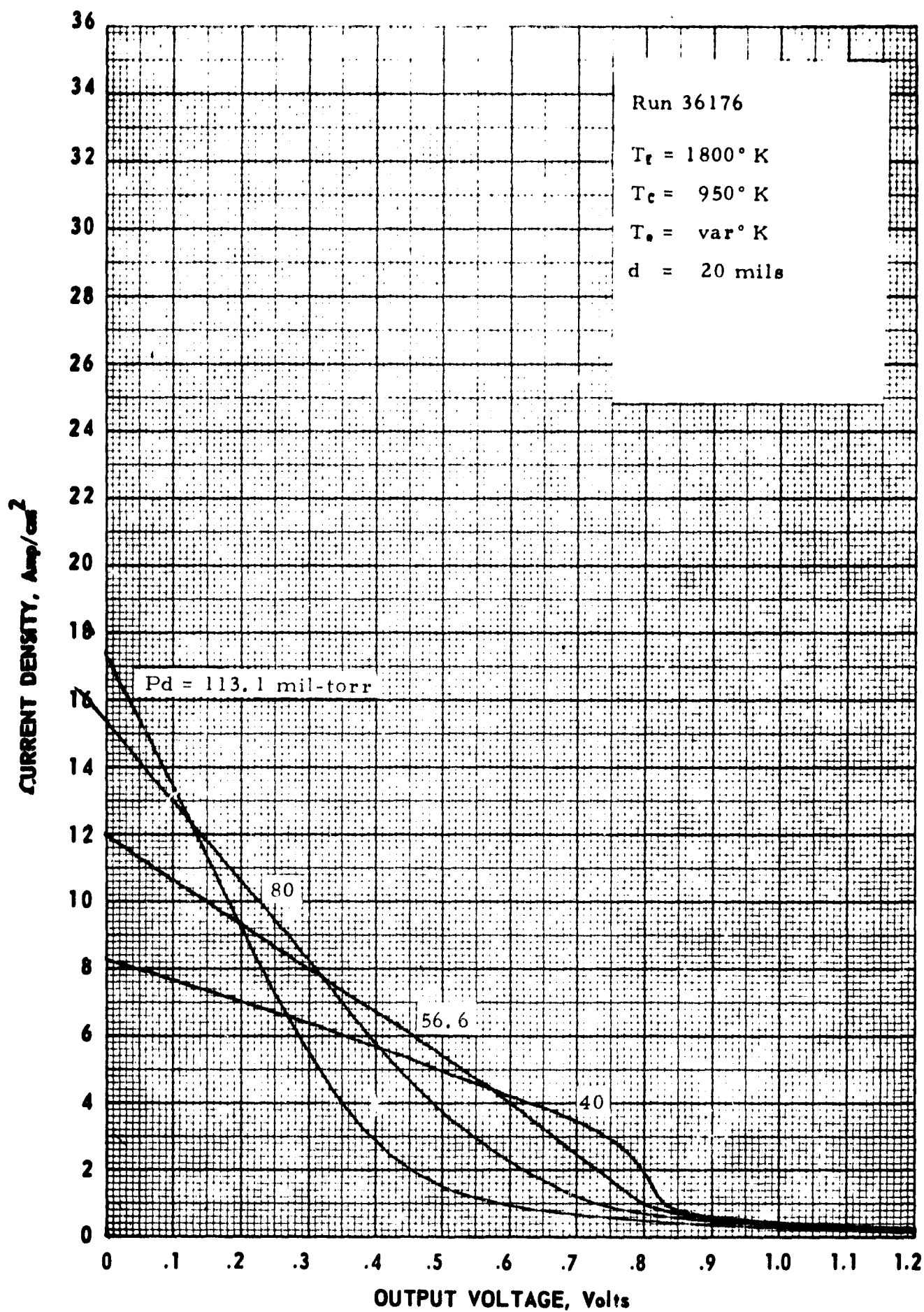


Figure III-23. Variable Cesium Pressure Family at $T_e = 1800^\circ \text{ K}$ and $d = 20 \text{ mils}$.

68-TR-11-16

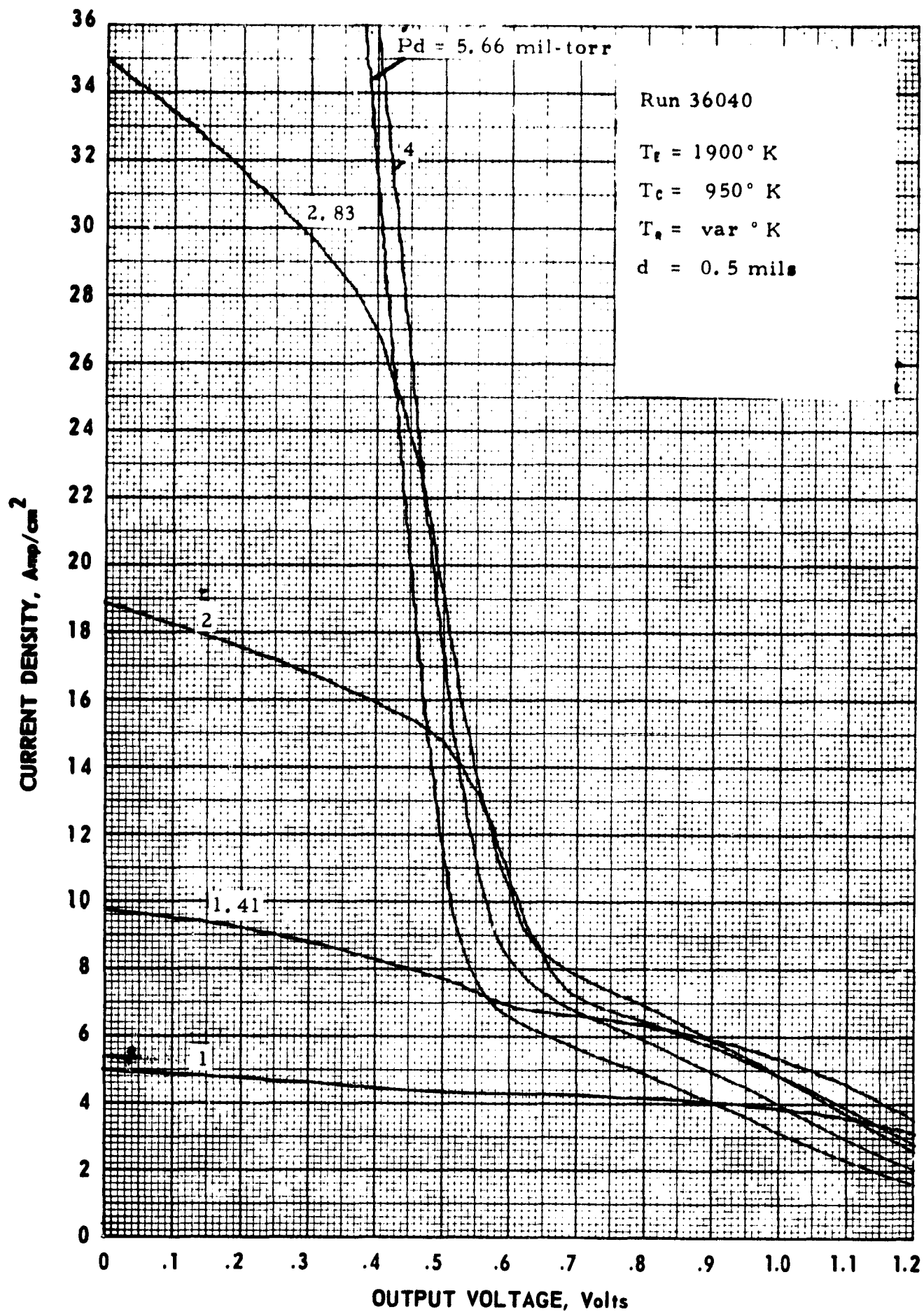


Figure III-24. Variable Cesium Pressure Family at T_f = 1900° K and d = 0.5 mils.



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68-TR-11-17

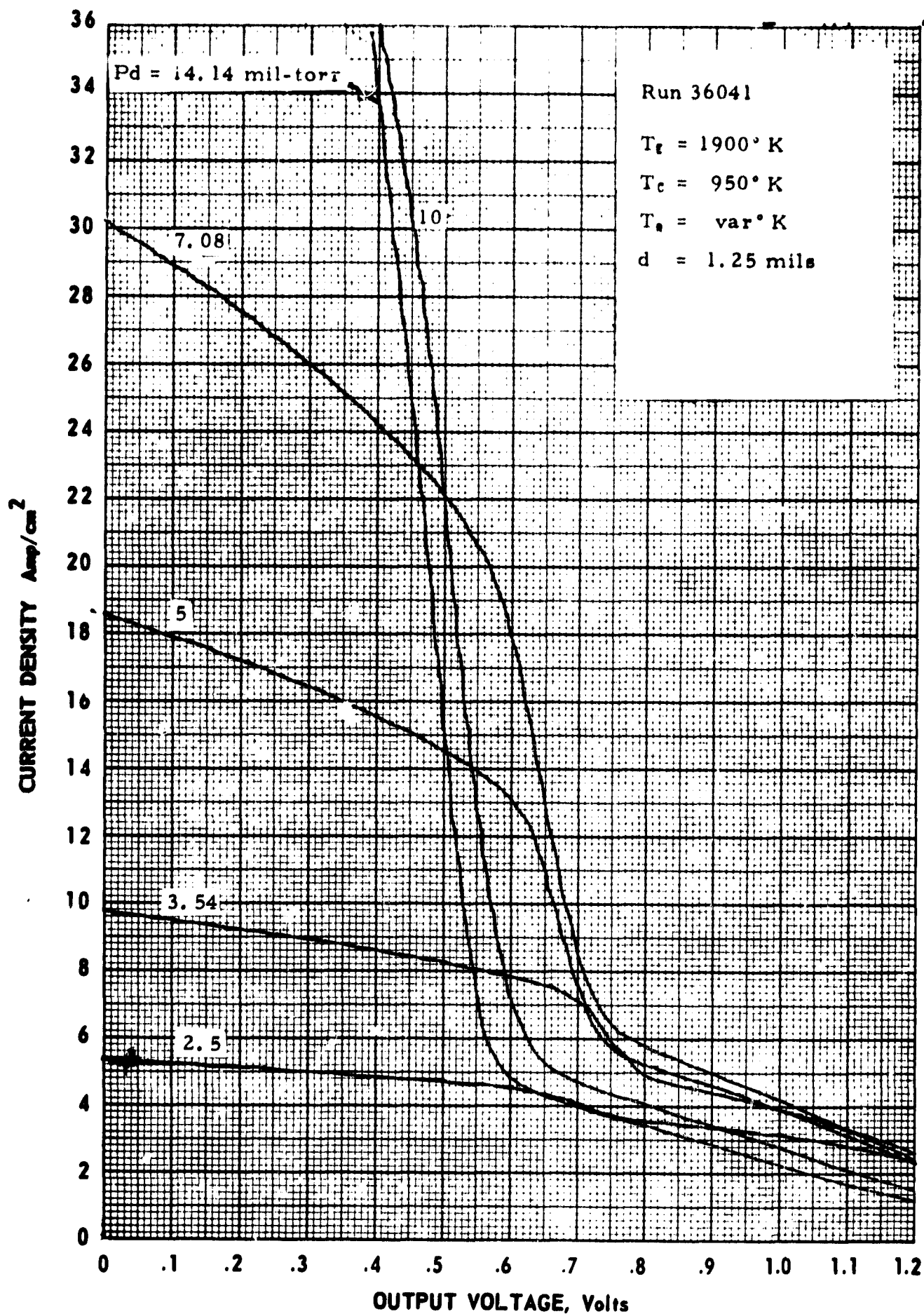


Figure III-25. Variable Cesium Pressure Family at $T_g = 1900^\circ \text{K}$ and $d = 1.25 \text{ mils}$.



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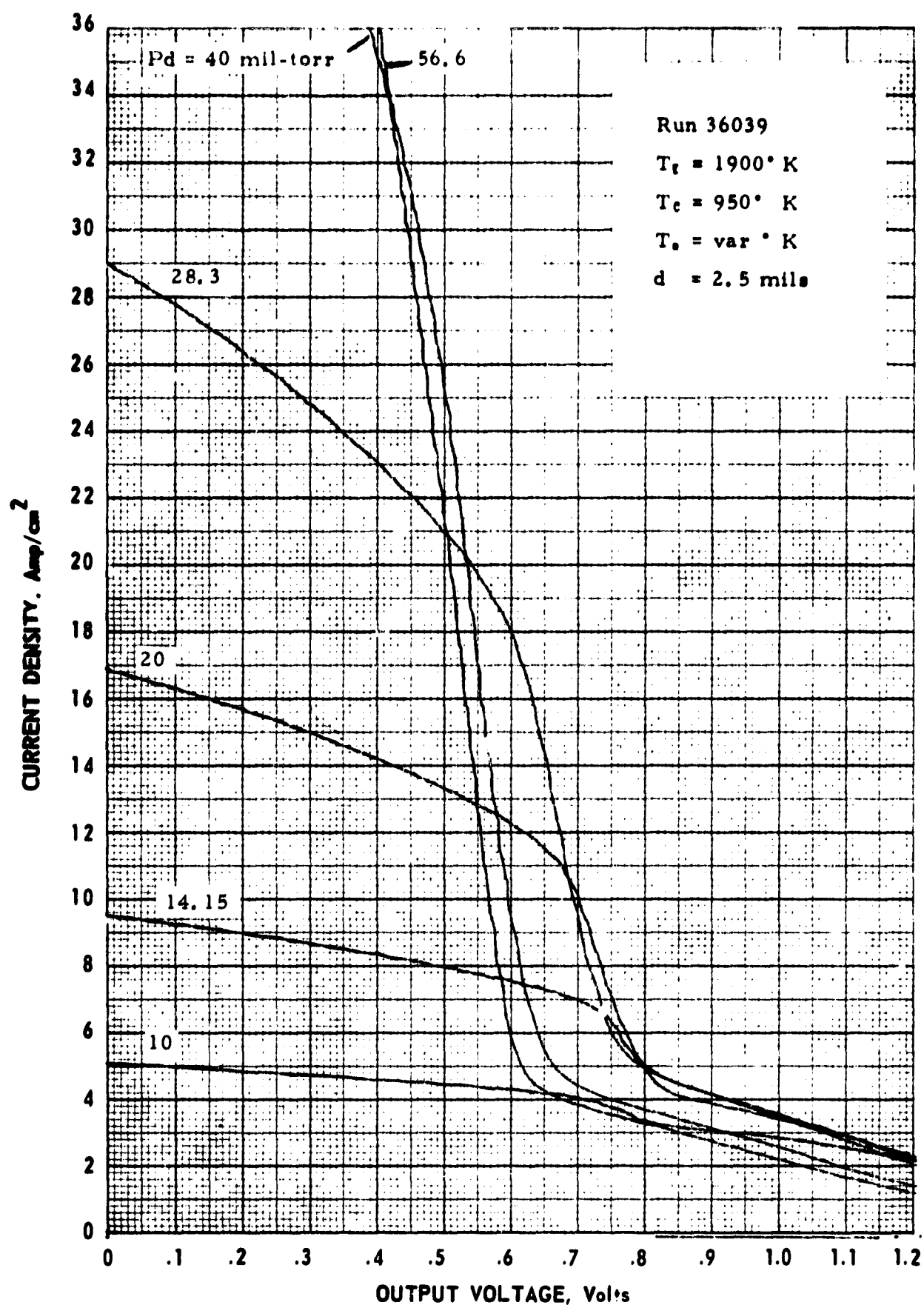


Figure III-26. Variable Cesium Pressure Family at $T_f = 1900^\circ \text{K}$ and $d = 2.5 \text{ mils}$.



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68-TR-10-32

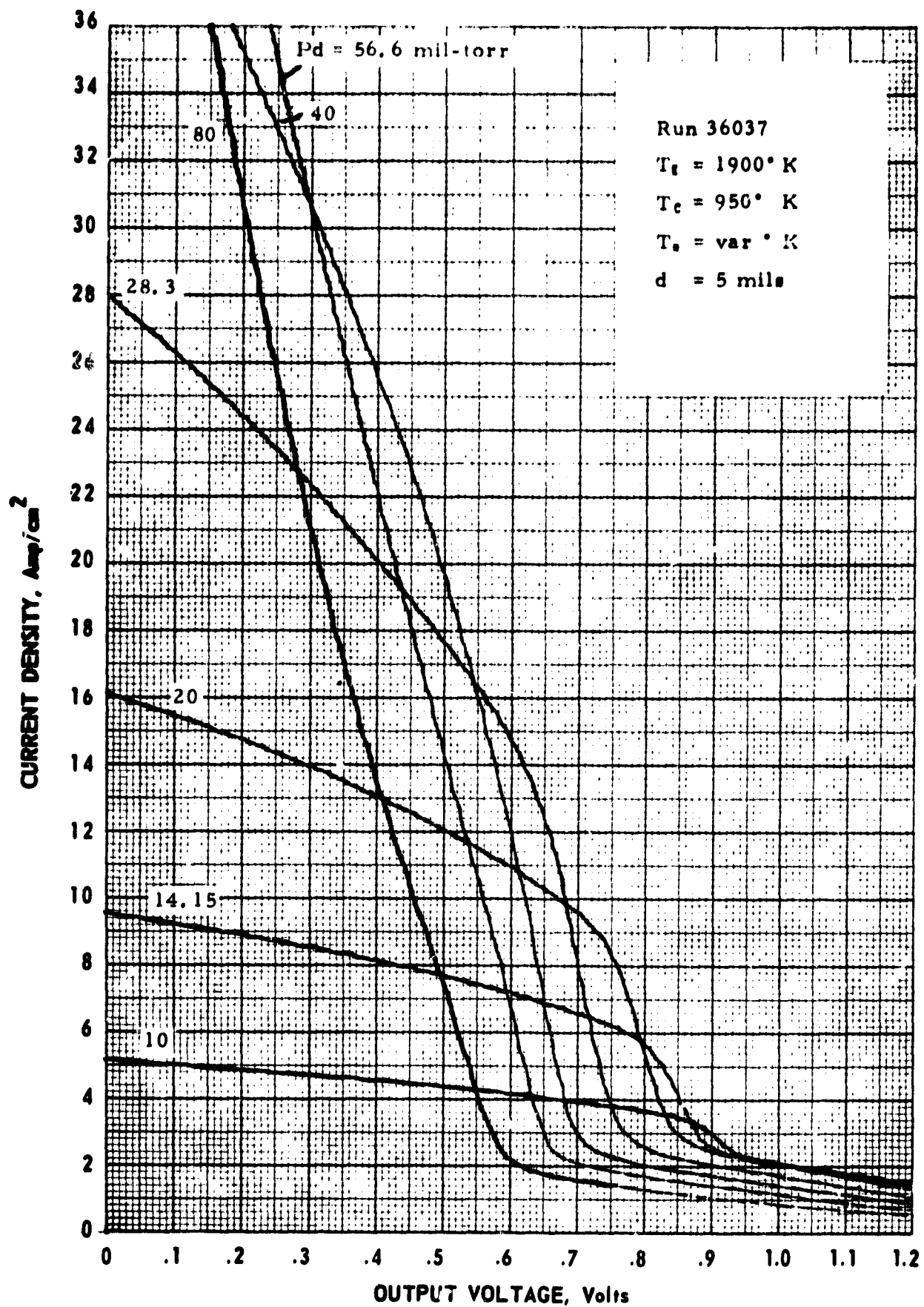


Figure III-27. Variable Cesium Pressure Family at $T_f = 1900^\circ K$ and $d = 5$ mils.

68-TR-10-35

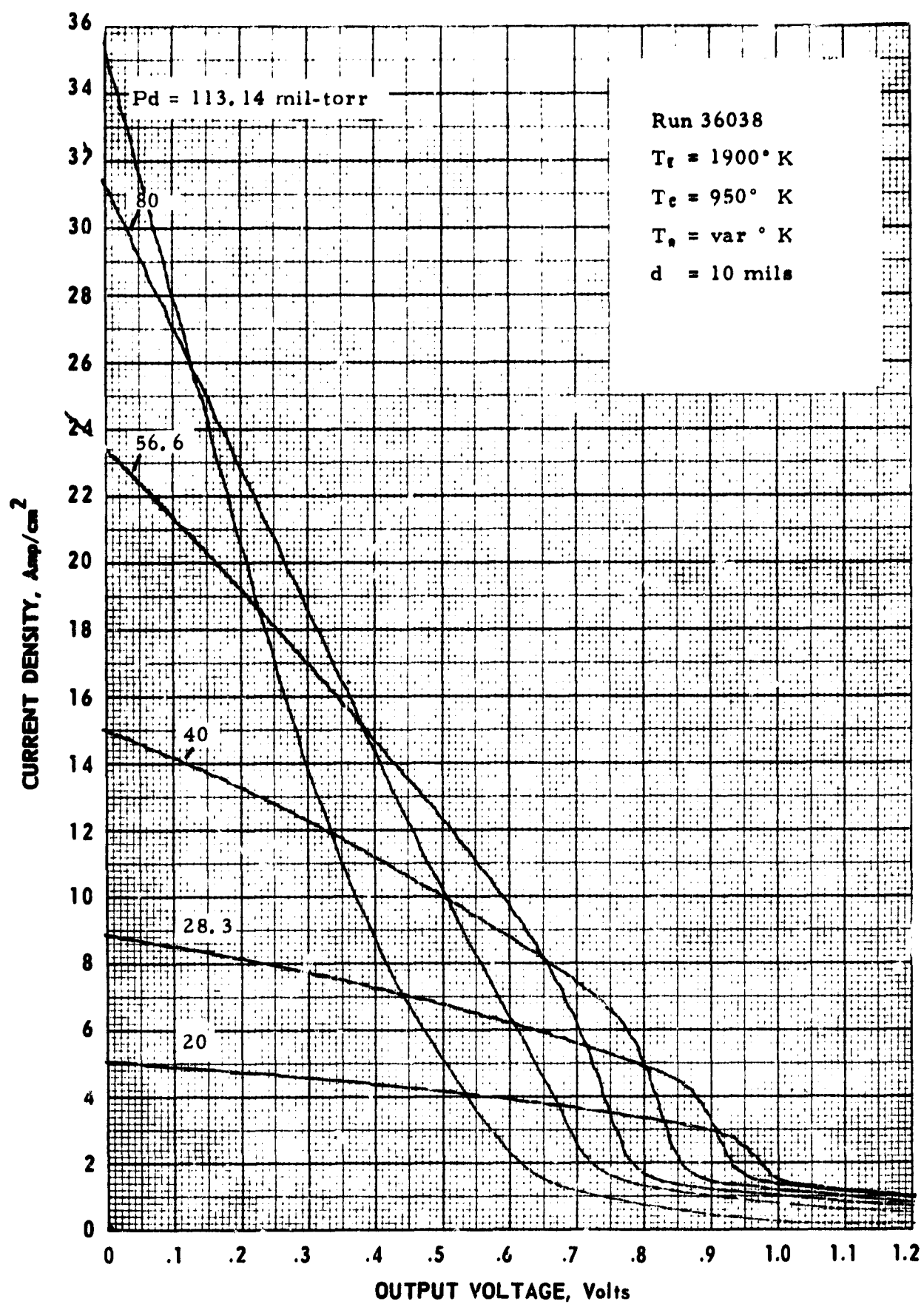


Figure III-28. Variable Cesium Pressure Family at $T_c = 1900^\circ \text{ K}$ and $d = 10 \text{ mils}$.

68-TR-11-18

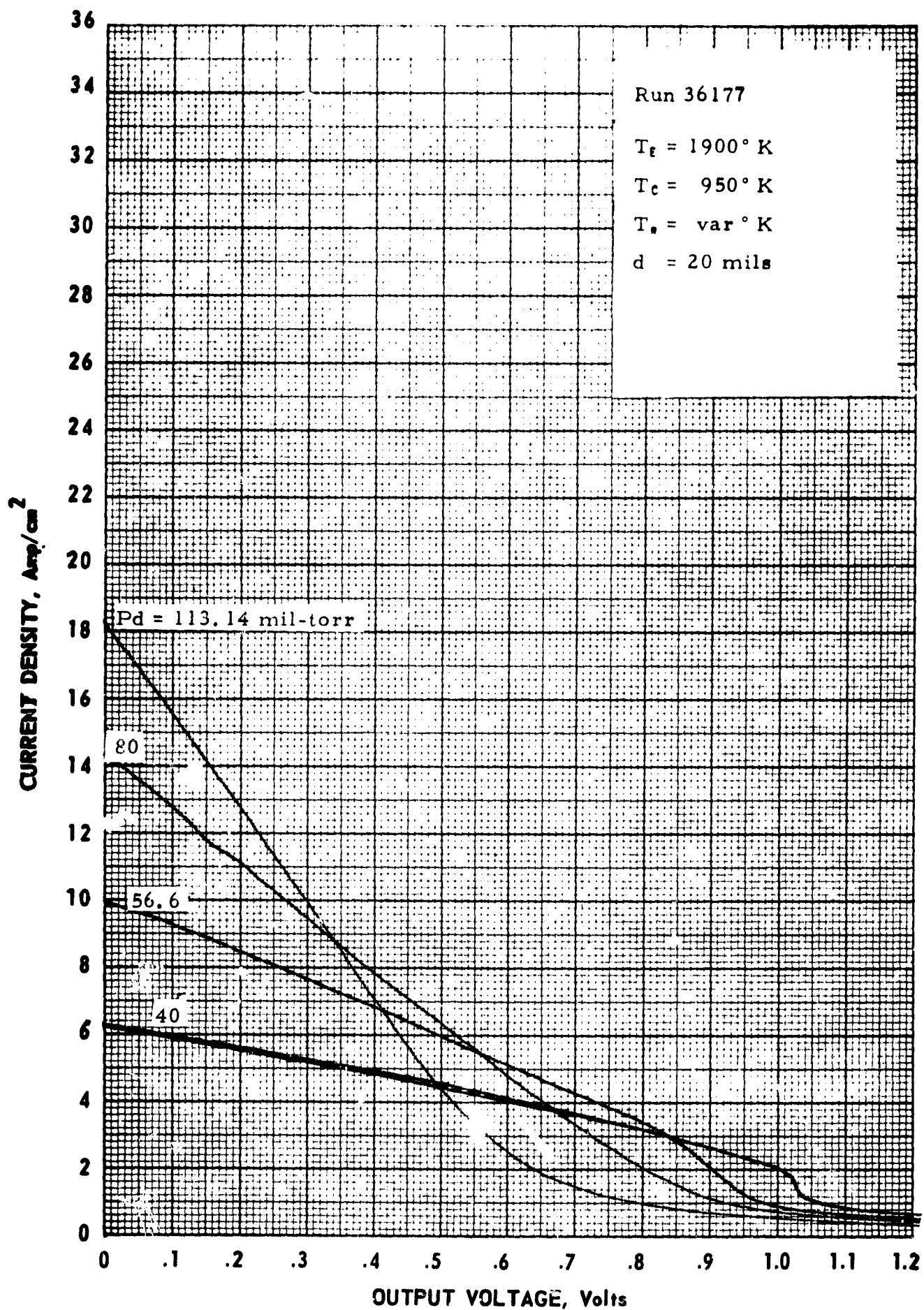


Figure III-29. Variable Cesium Pressure Family at $T_e = 1900^\circ \text{ K}$ and $d = 20 \text{ mils}$.

68-TR-10-52

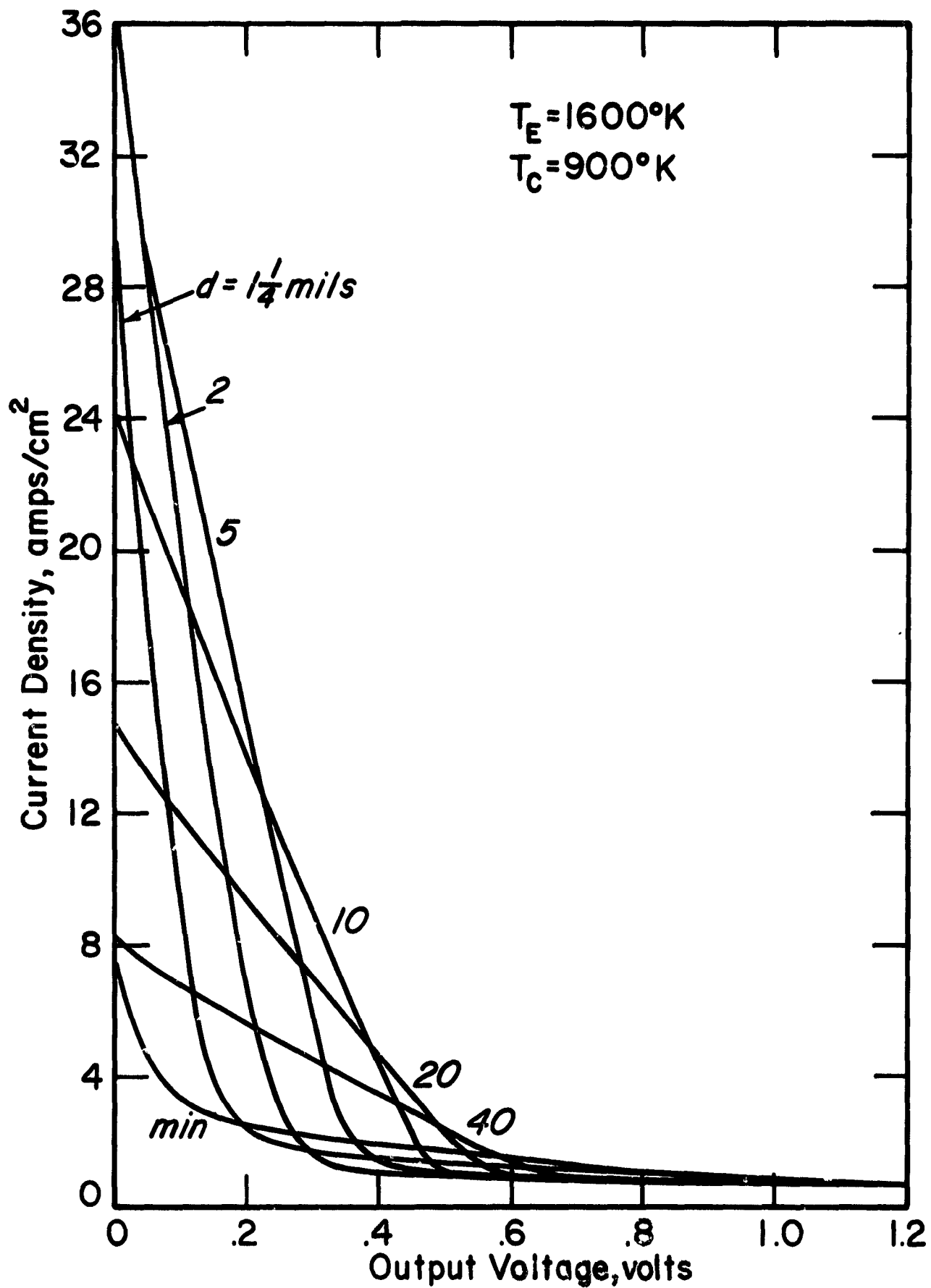


Figure III-30. Cesium Optimized Performance for Several Interelectrode Spacings at $T_E = 1600^\circ\text{K}$.

68-TR-10-14

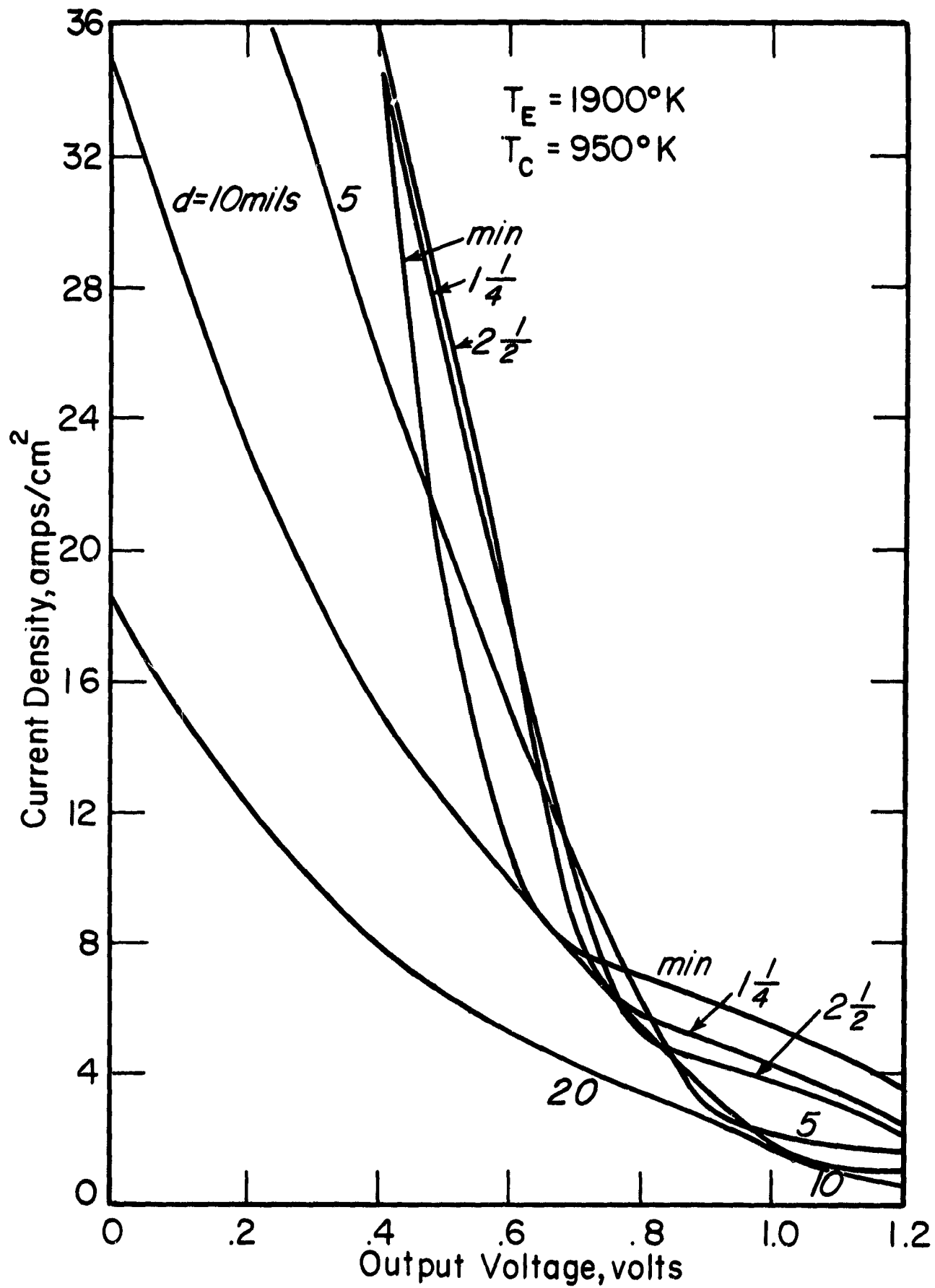


Figure III-31. Cesium Optimized Performance for Several Interelectrode Spacings at $T_E = 1900^\circ\text{K}$.

68-TR-10-11

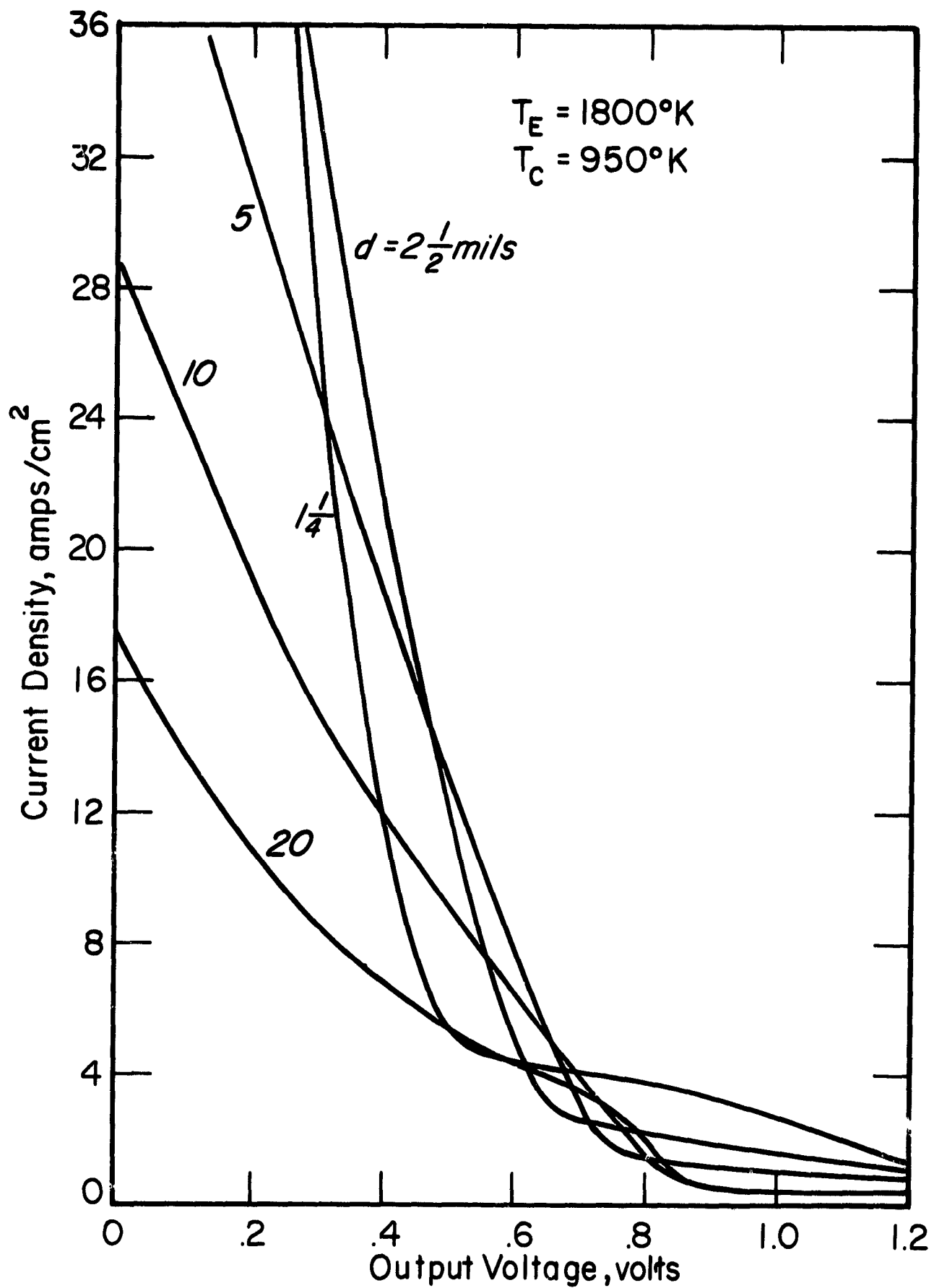


Figure III-32. Cesium Optimized Performance for Several Interelectrode Spacings at $T_c = 1800^\circ\text{K}$.

68-TR-10-13

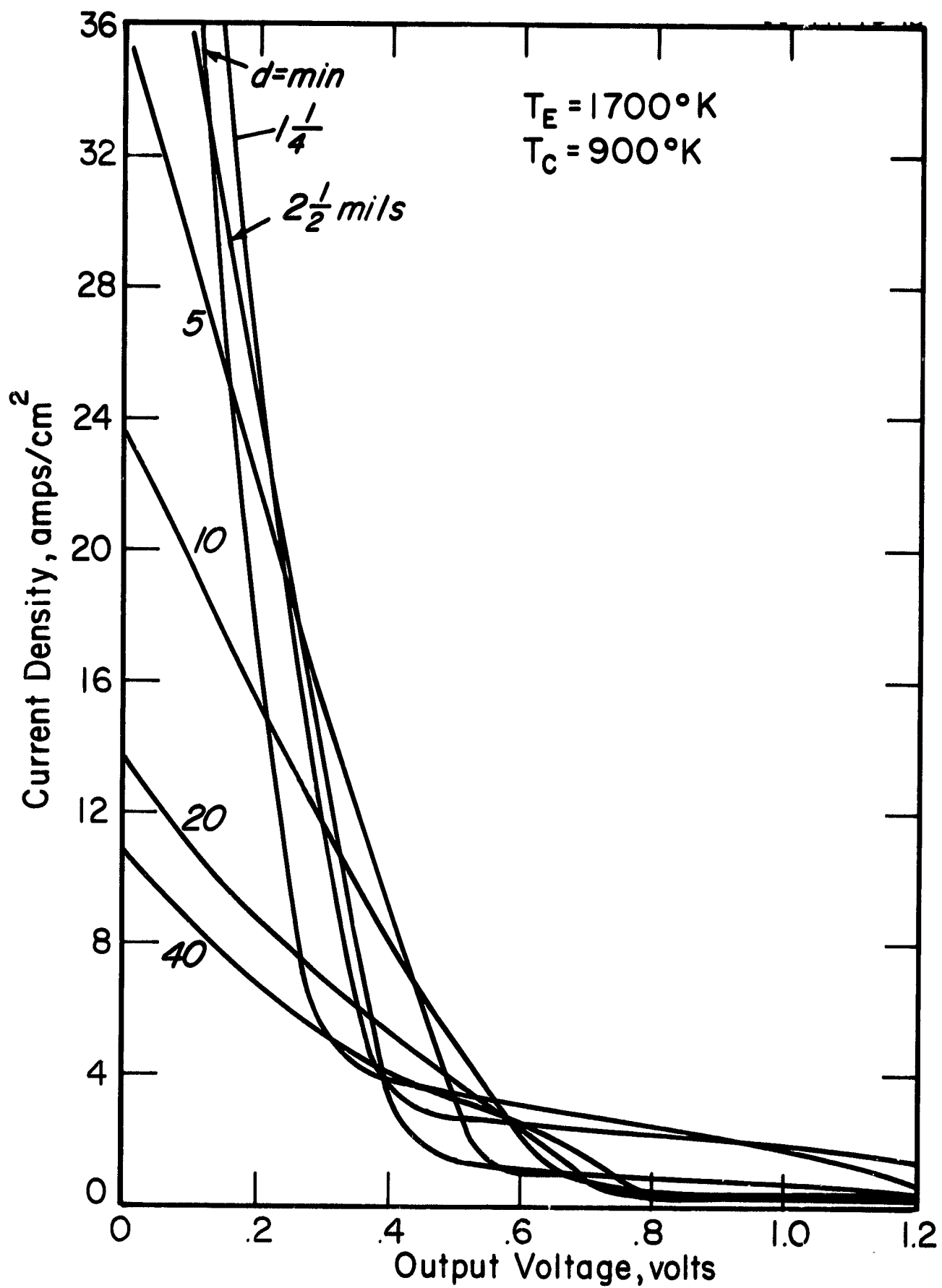


Figure III-33. Cesium Optimized Performance for Several Interelectrode Spacings at $T_E = 1700^\circ\text{K}$.



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68-TR-10-12

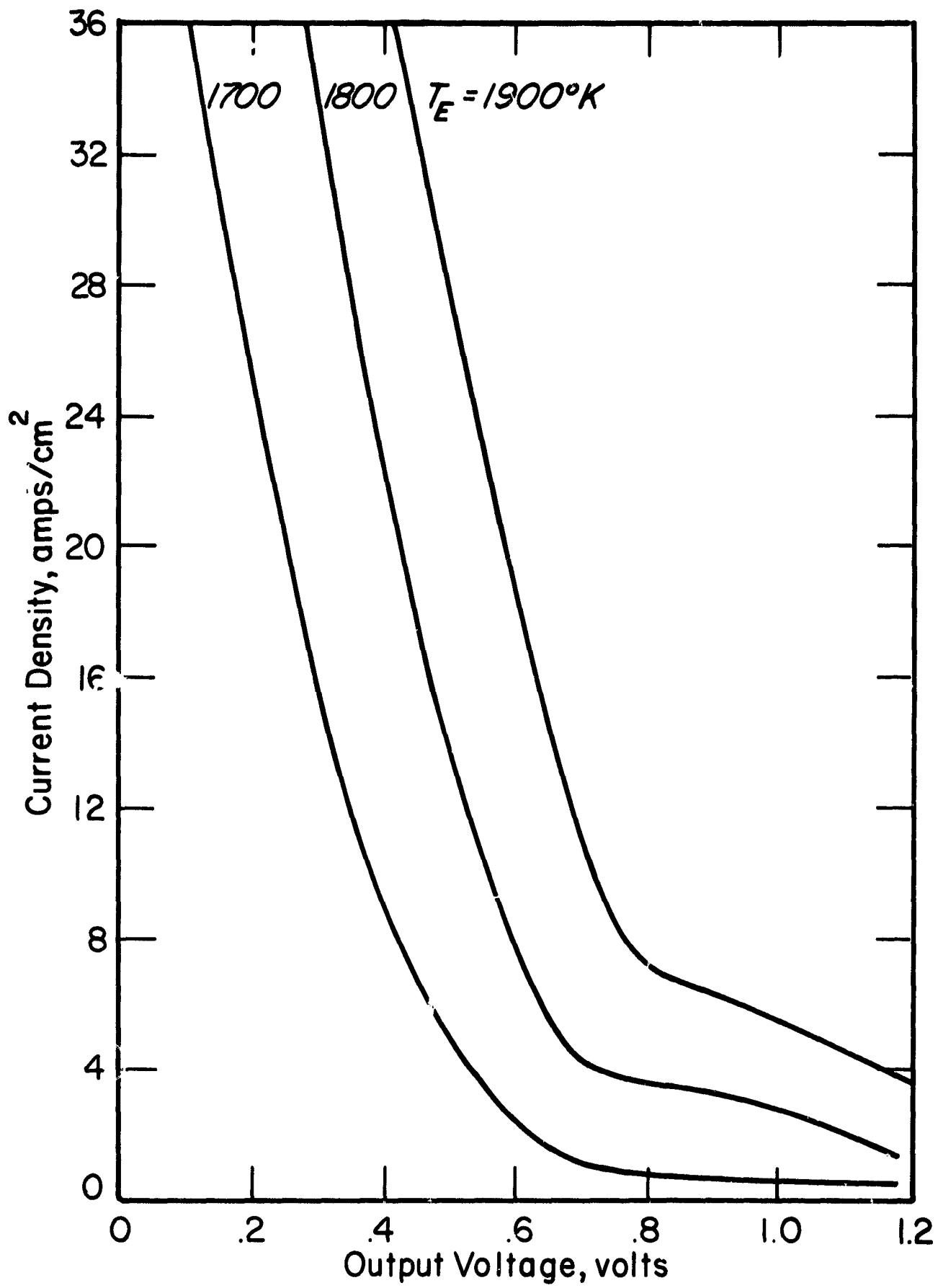


Figure III-34. Fully Optimized Performance at Several Emitter Temperatures.

68-TR-10-43

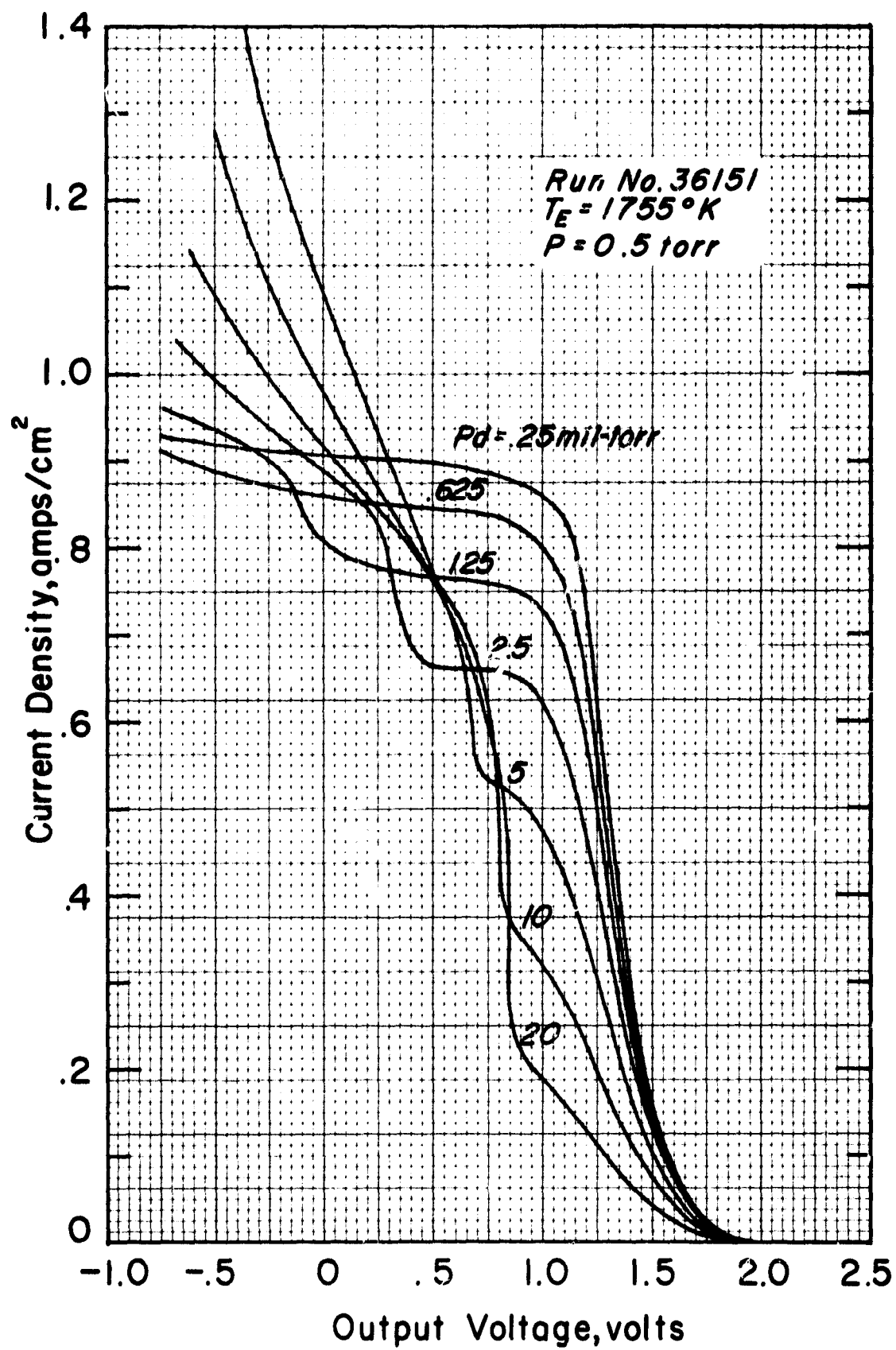


Figure III-35. Variable Spacing Family at $\beta = 10$ and $P = 0.5 \text{ torr}$.



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68-TR-10-51

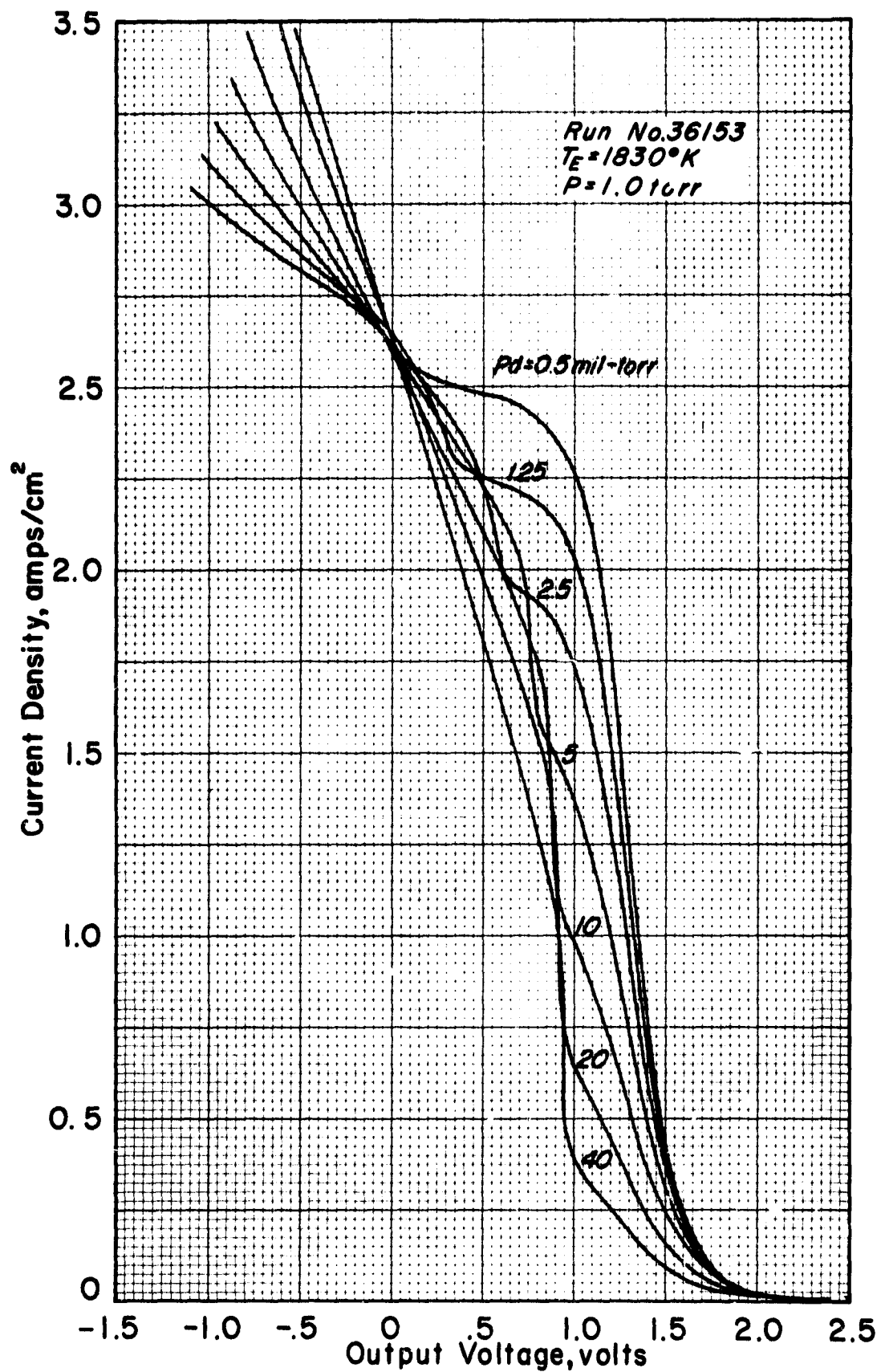


Figure III-36. Variable Spacing Family at $\beta = 10$ and $P = 1 \text{ torr}$.

68-TR-10-46

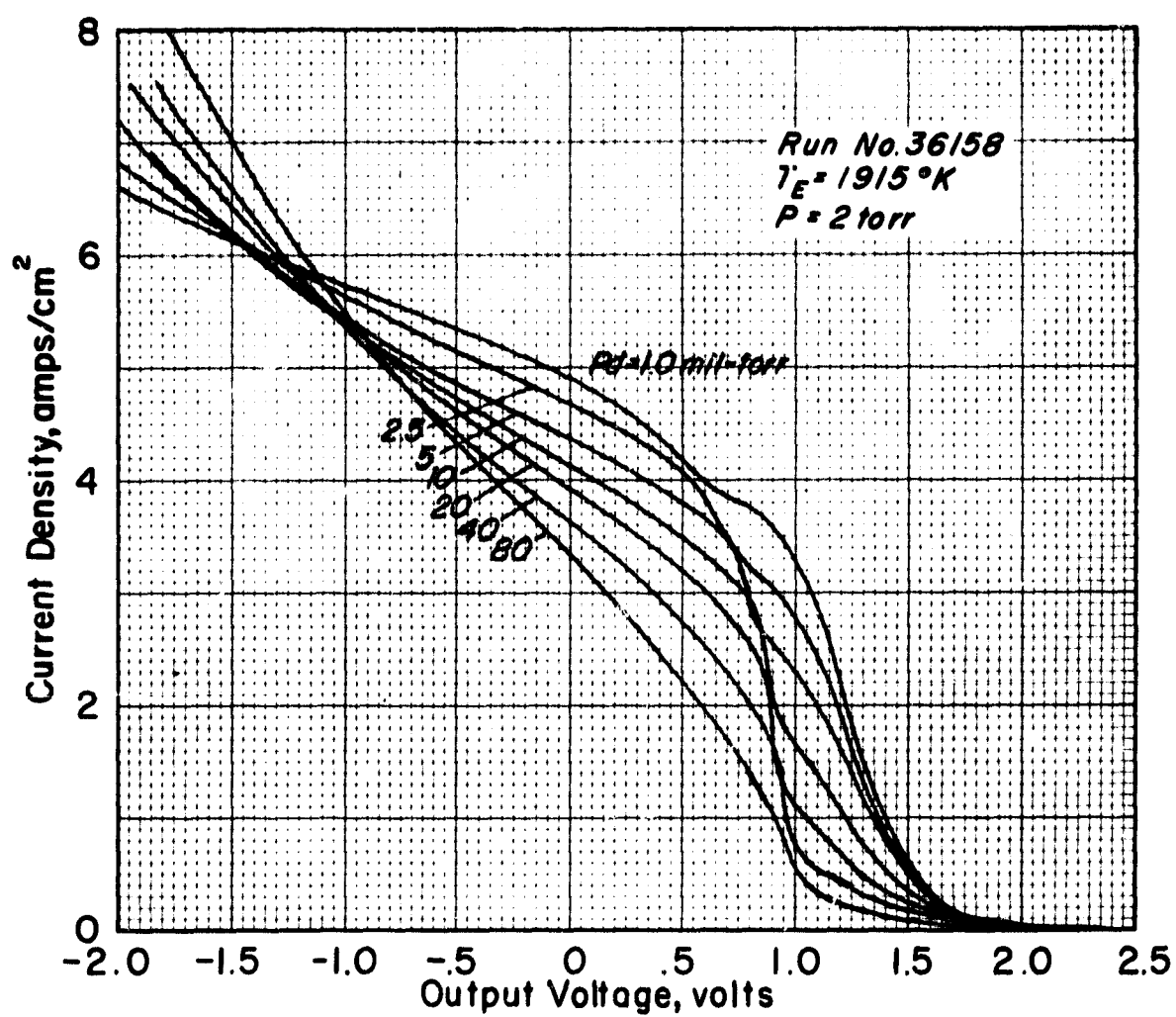


Figure III-37. Variable Spacing Family at $\beta = 10$ and $P = 2$ torr.



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68-TR-10-42

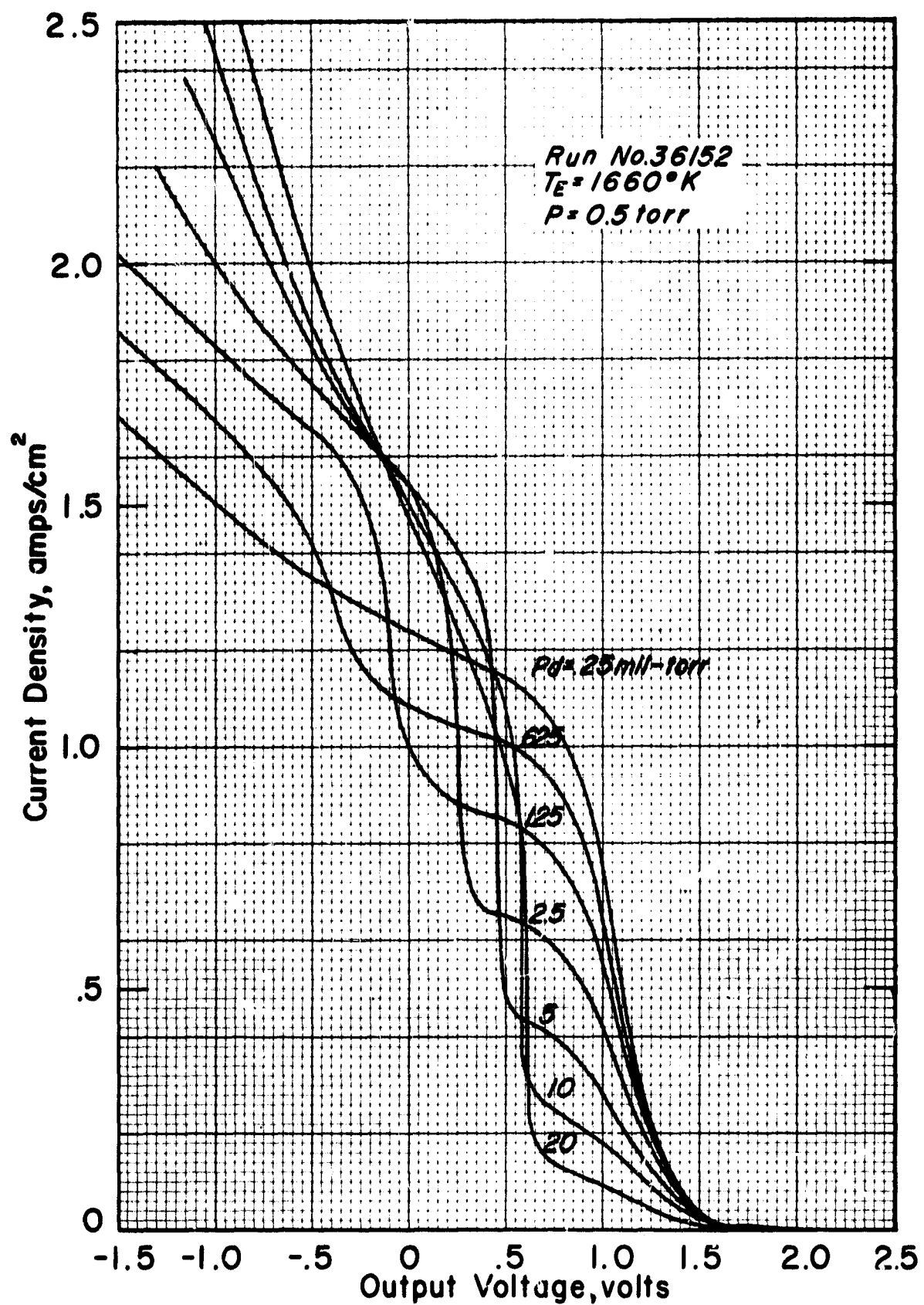


Figure III-38. Variable Spacing Family at $\beta = 1$ and $P = 0.5 \text{ torr}$.

68-TR-10-48

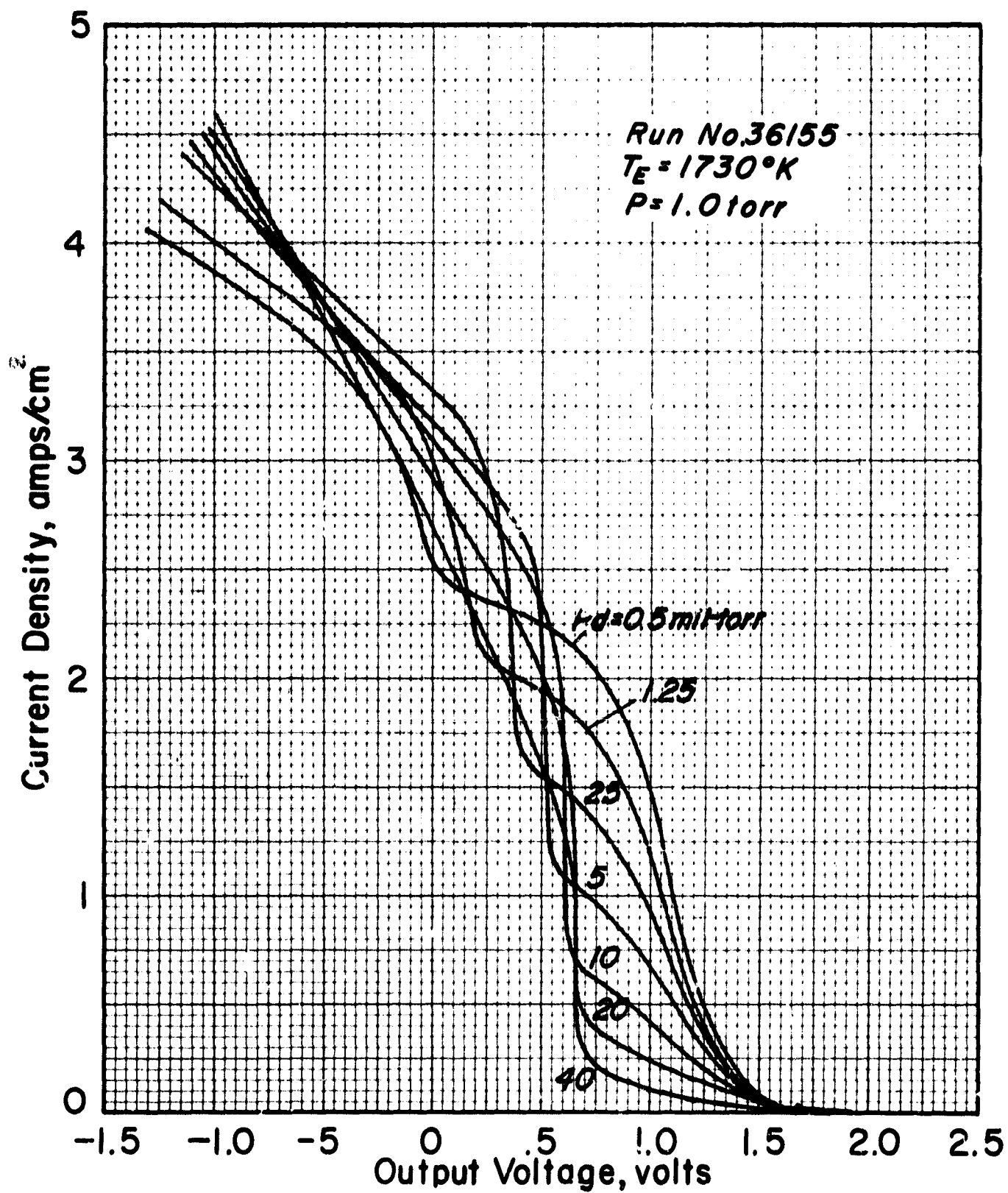


Figure III-39. Variable Spacing Family at $\beta = 1$ $P = 1 \text{ torr}$.

68-TR-10-45

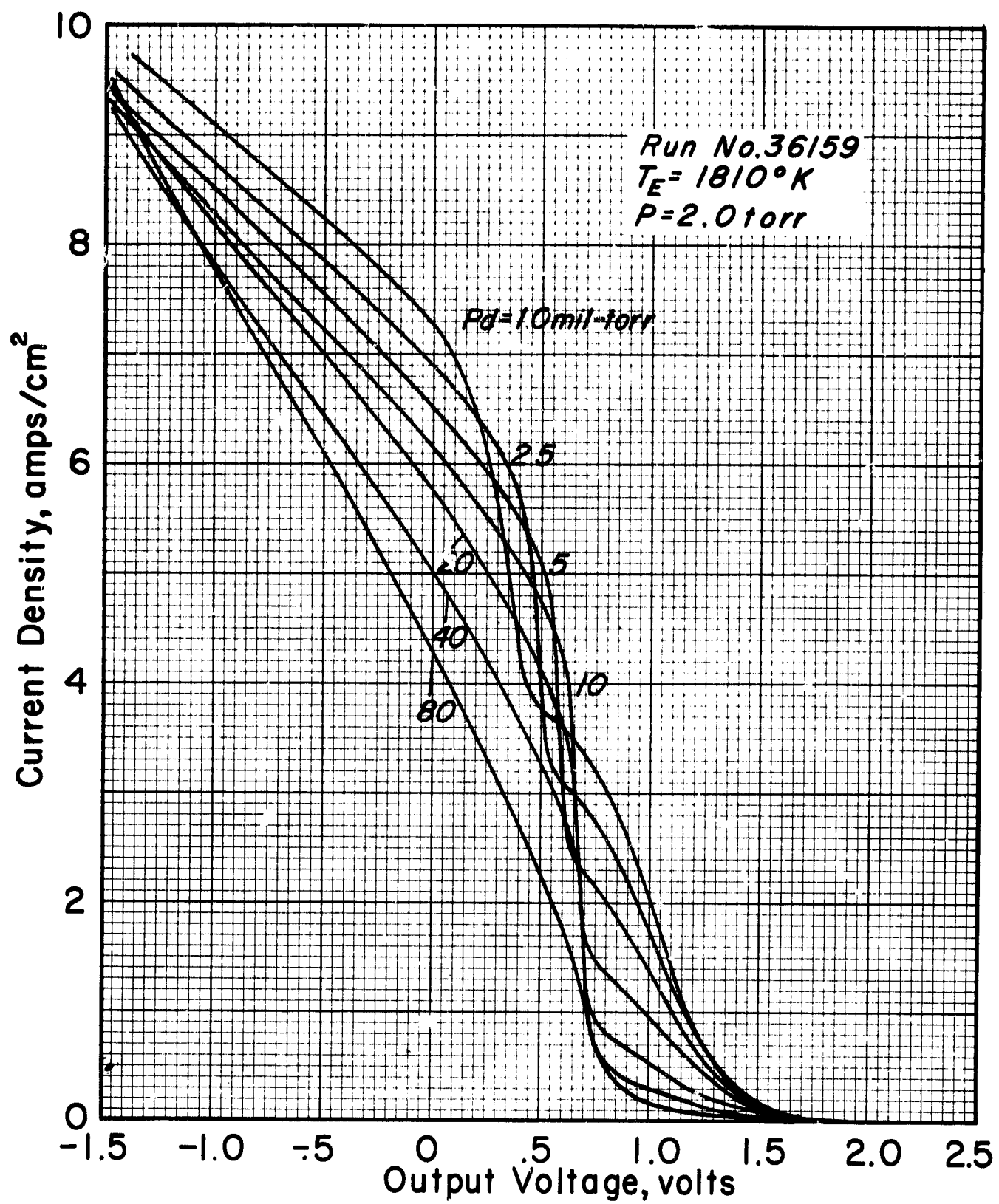


Figure III-40. Variable Spacing Family at $\beta = 1$ and $P = 2$ torr.

68-TR-10-49

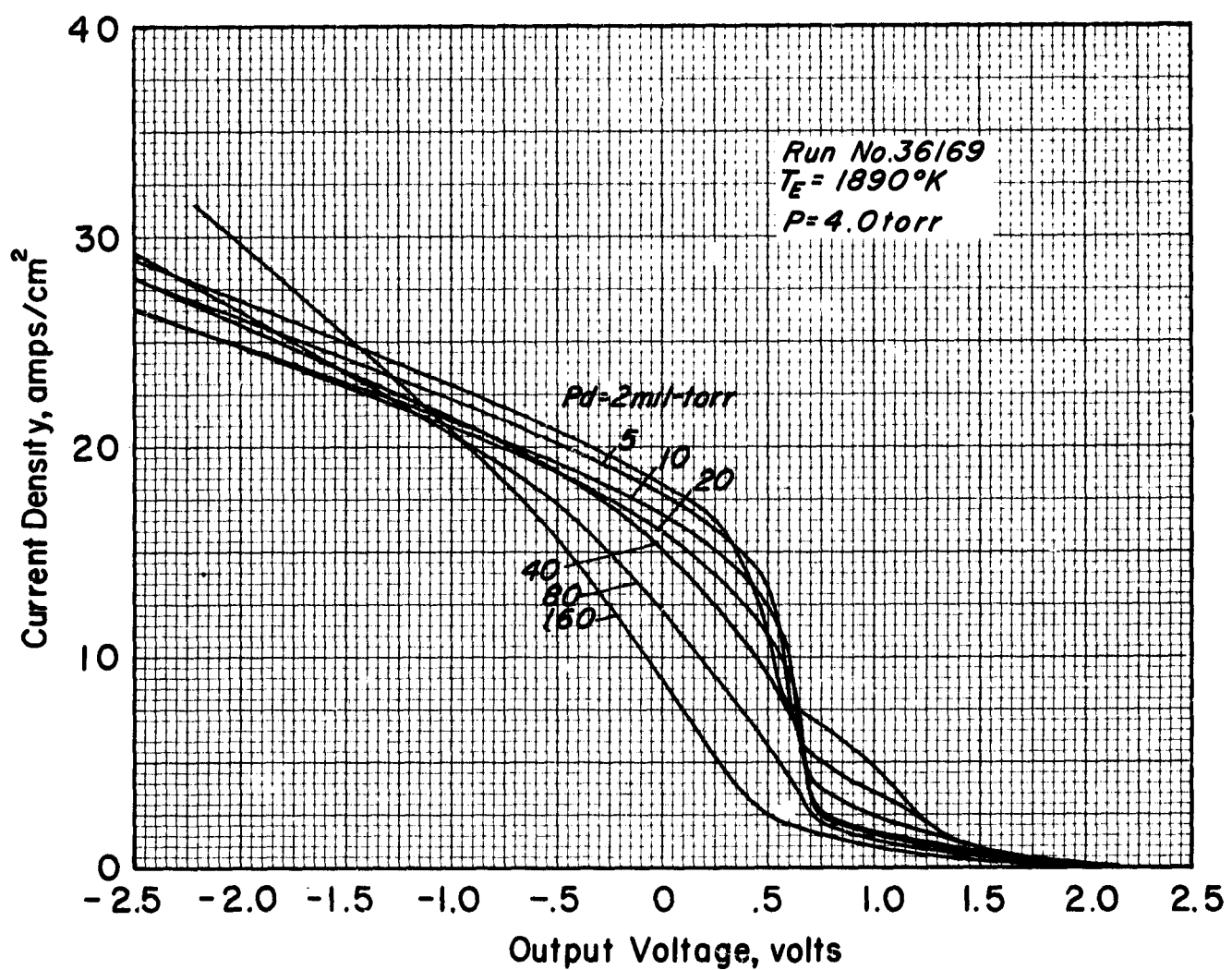


Figure III-41. Variable Spacing Family at $\beta = 1$ and $P = 4 \text{ torr}$.



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68-TR-10-47

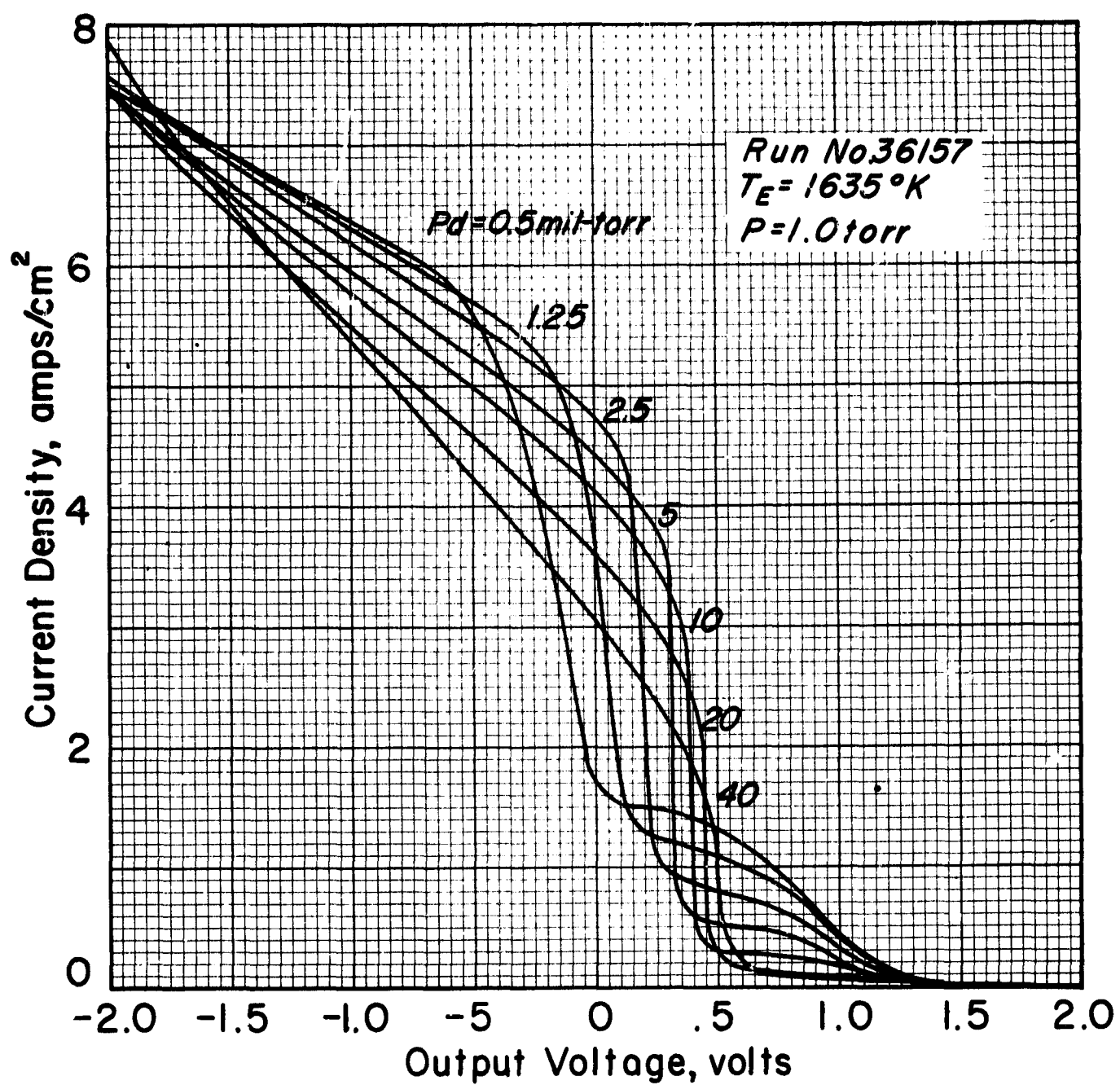


Figure III-42. Variable Spacing Family at $\beta = 0.1$ and $P = 1$ torr.

68-TR-10-44

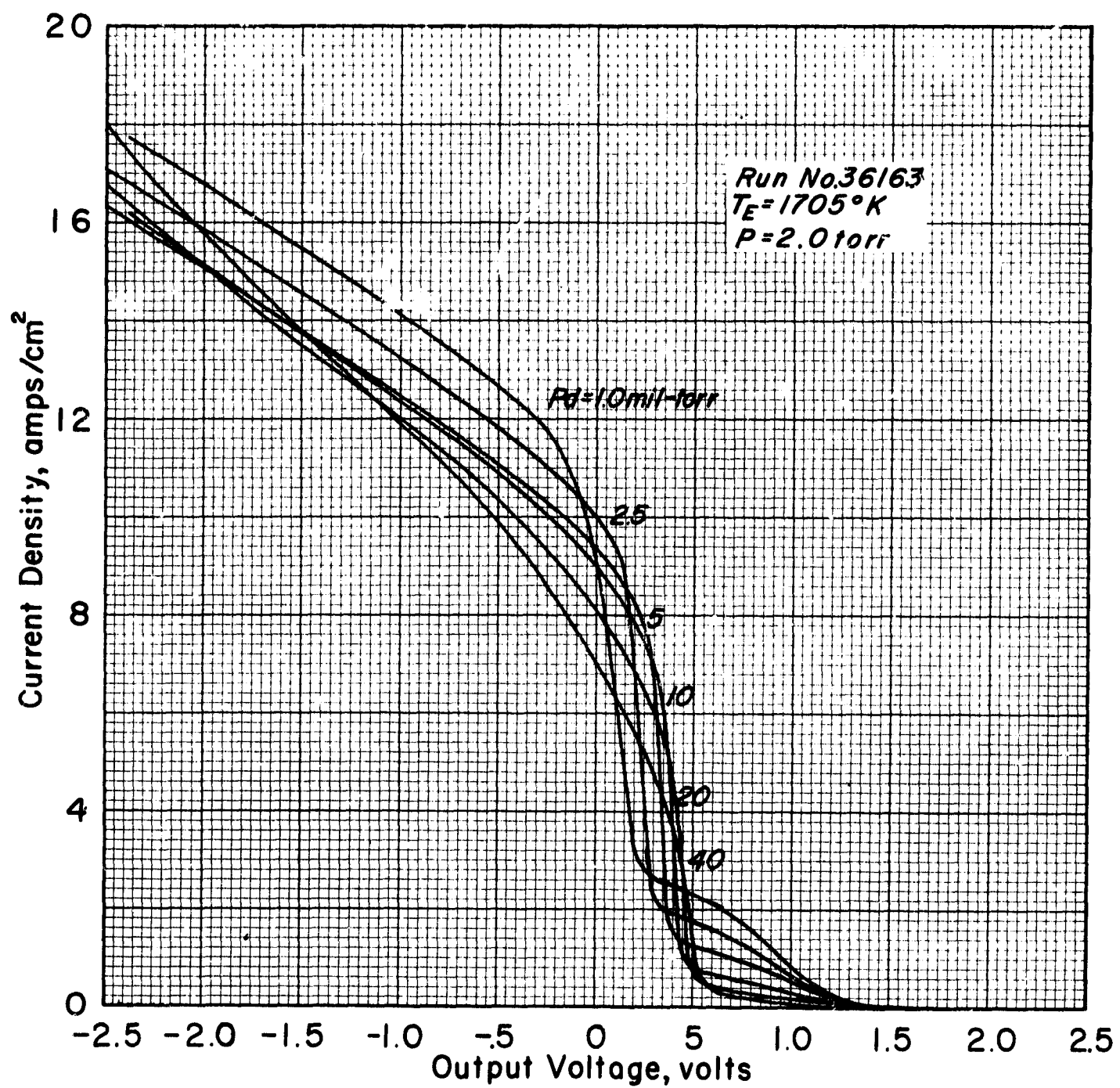


Figure III-43, Variable Spacing Family at $\beta = 0.1$ and $P = 2$ torr.

68-TR-10-50

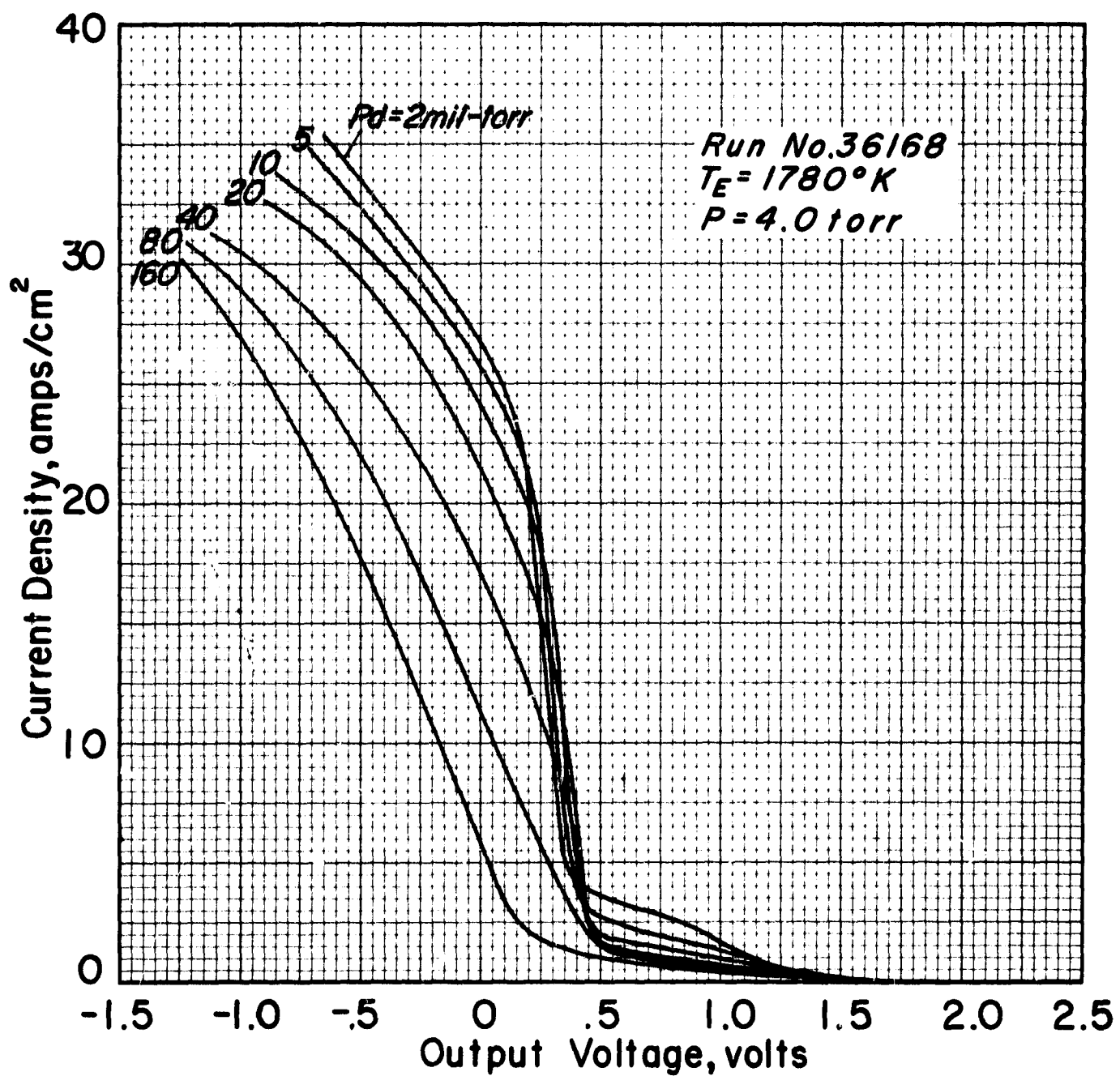


Figure III-44, Variable Spacing Family at $\beta = 0.1$ and $P = 4$ torr.

68-TR-10-28

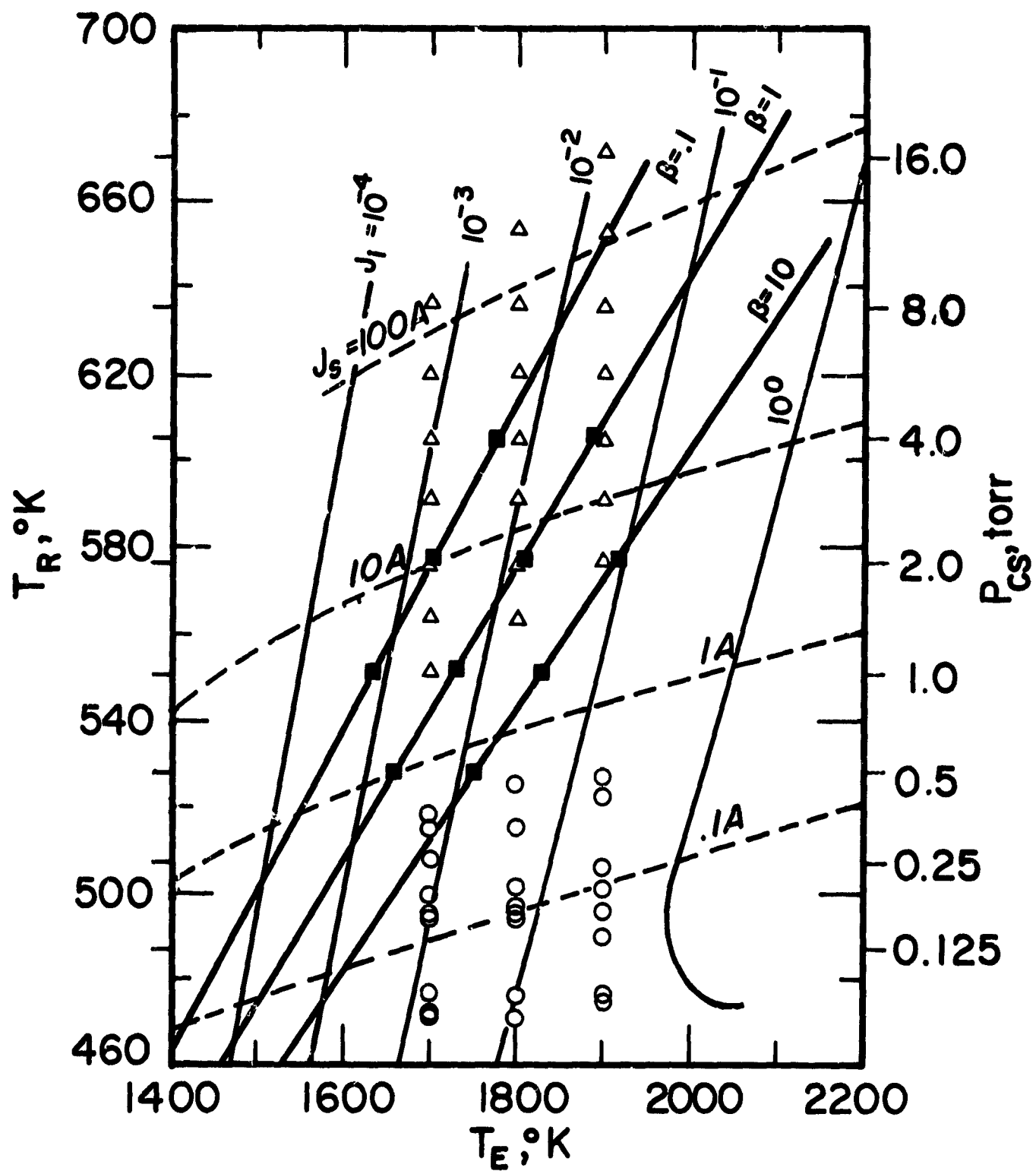


Figure III-45. Emission Map of the Emitter Showing Location of the Data Points.

- Work Function Data
- △ Cesium Families
- Spacing Family

68-TR-10-58

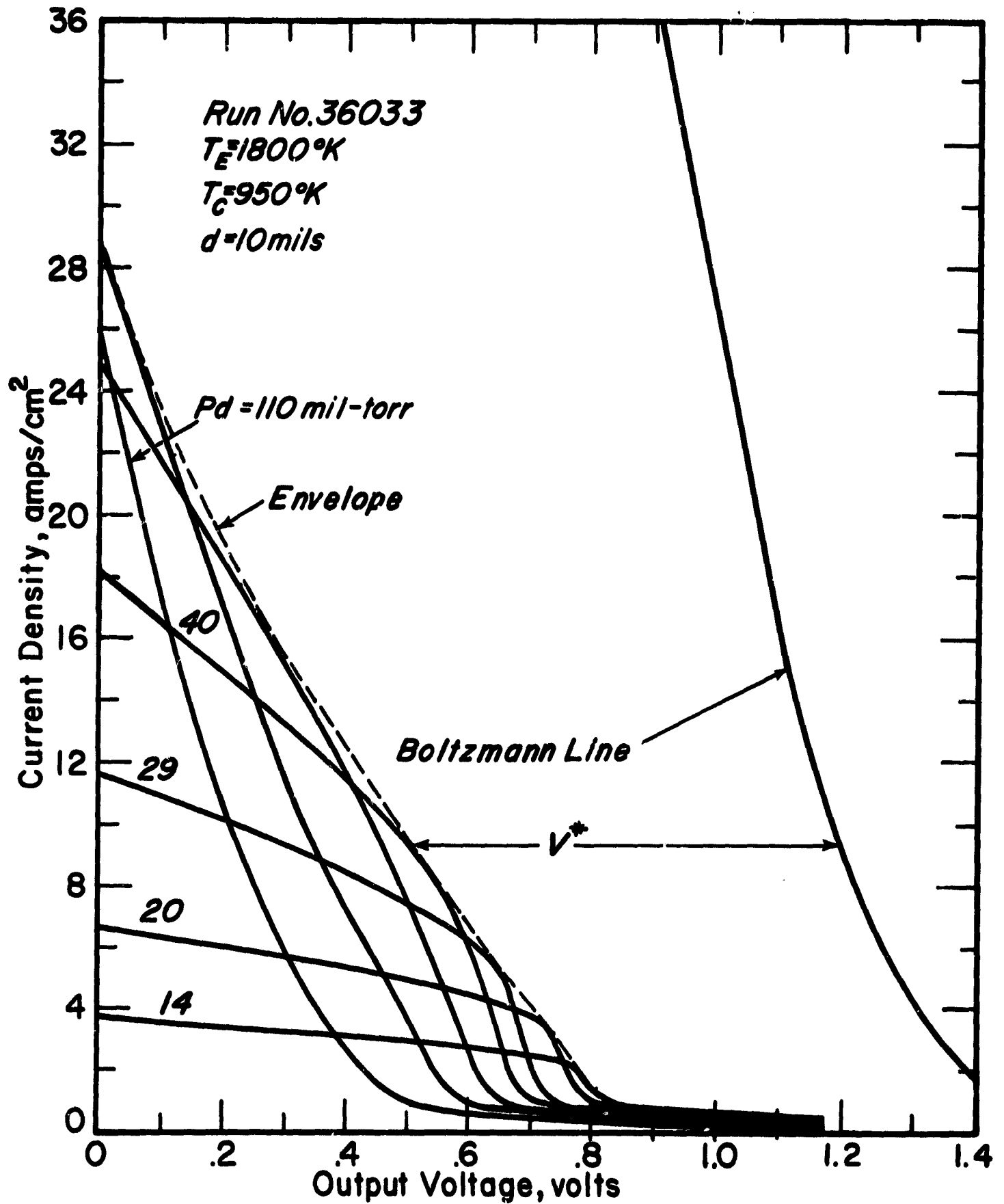


Figure III-46. Cesium Family Illustrating Deviation from Ideal Performance.

68-TR-10-57

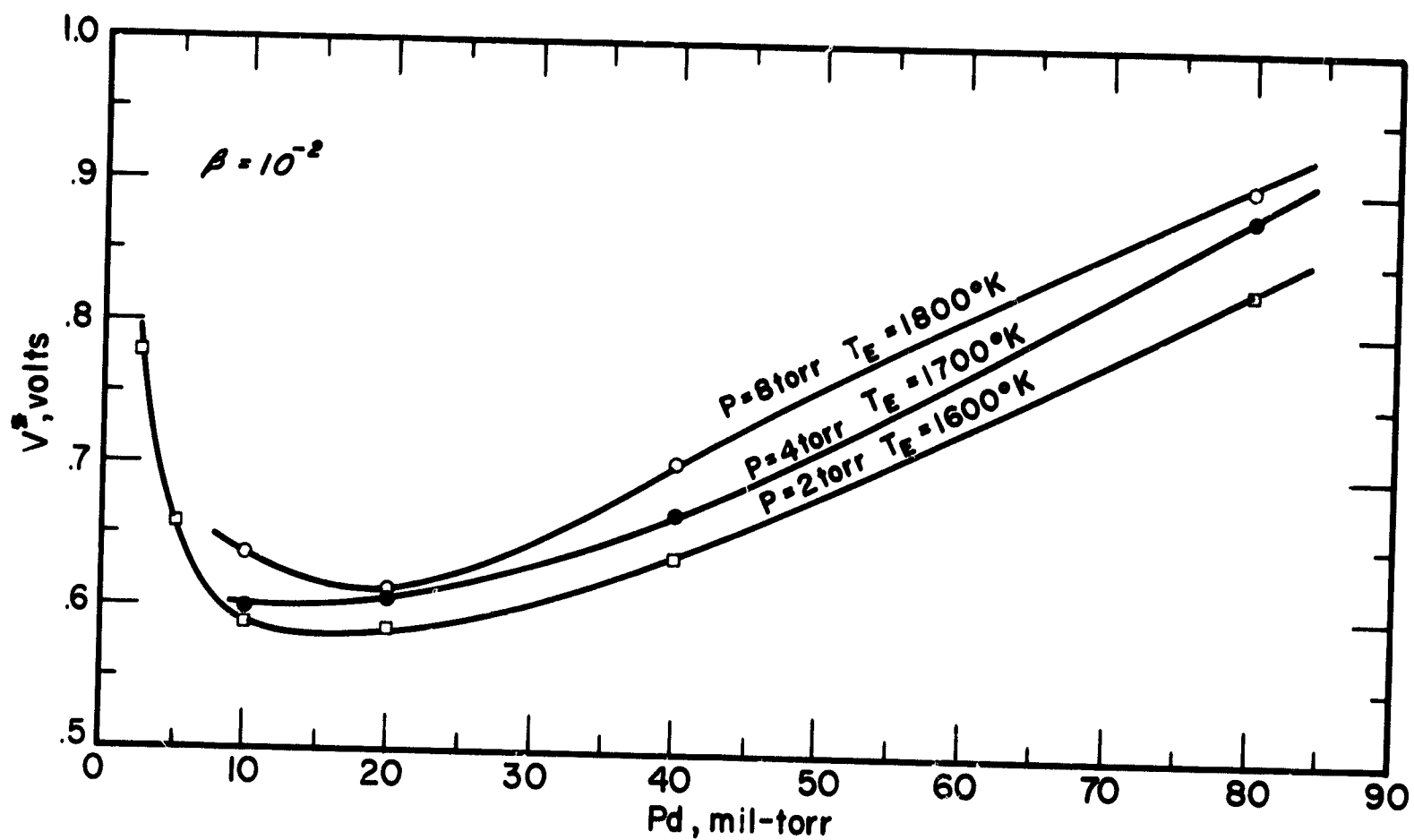


Figure III-47. Voltage Loss Correlation for Several Cesium Pressures at $\beta = 10^{-2}$.

68-TR-10-56

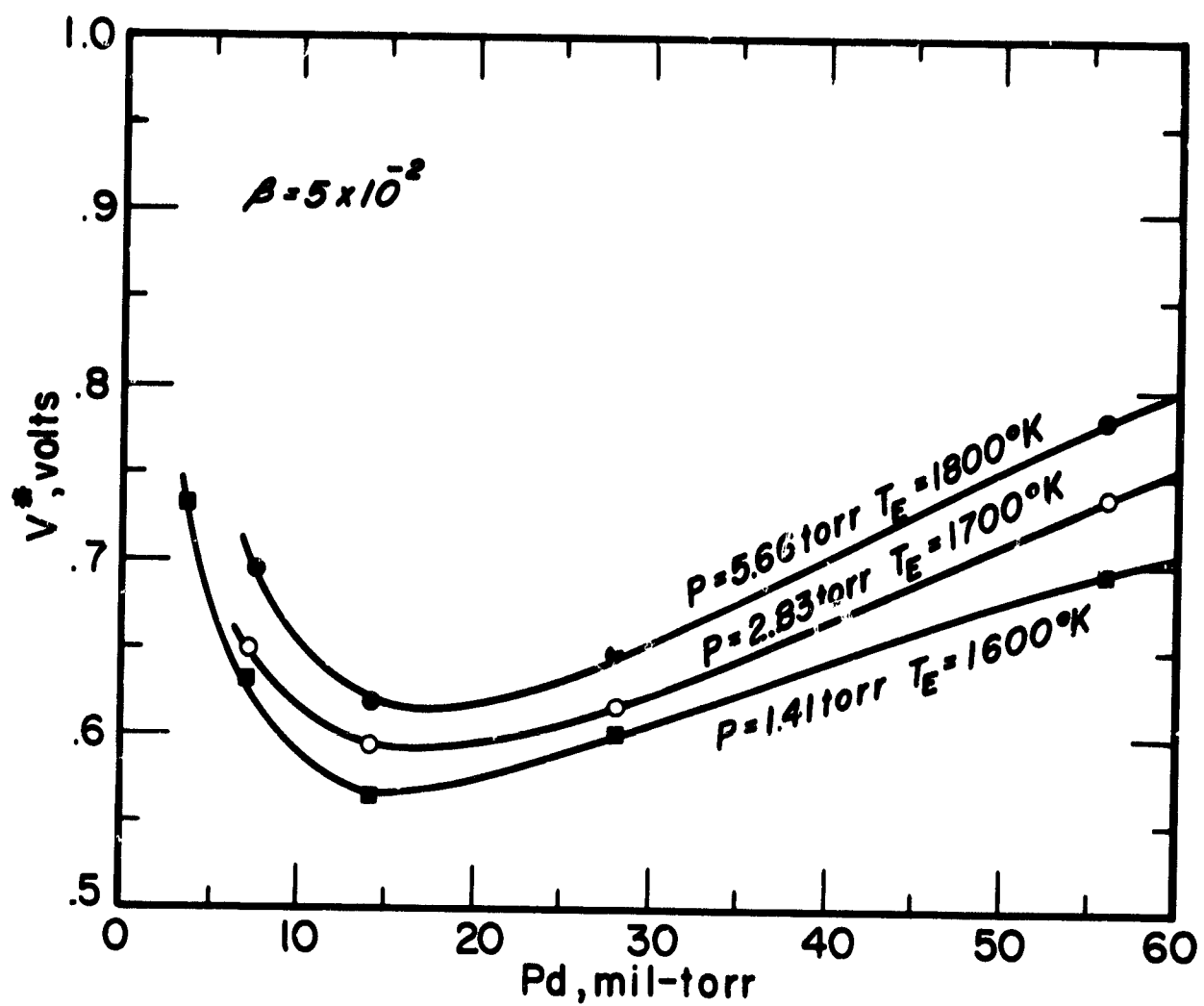


Figure III-48. Voltage Loss Correlation for Several Cesium Pressures at $\beta = 5 \times 10^{-1}$.

68-TR-10-55

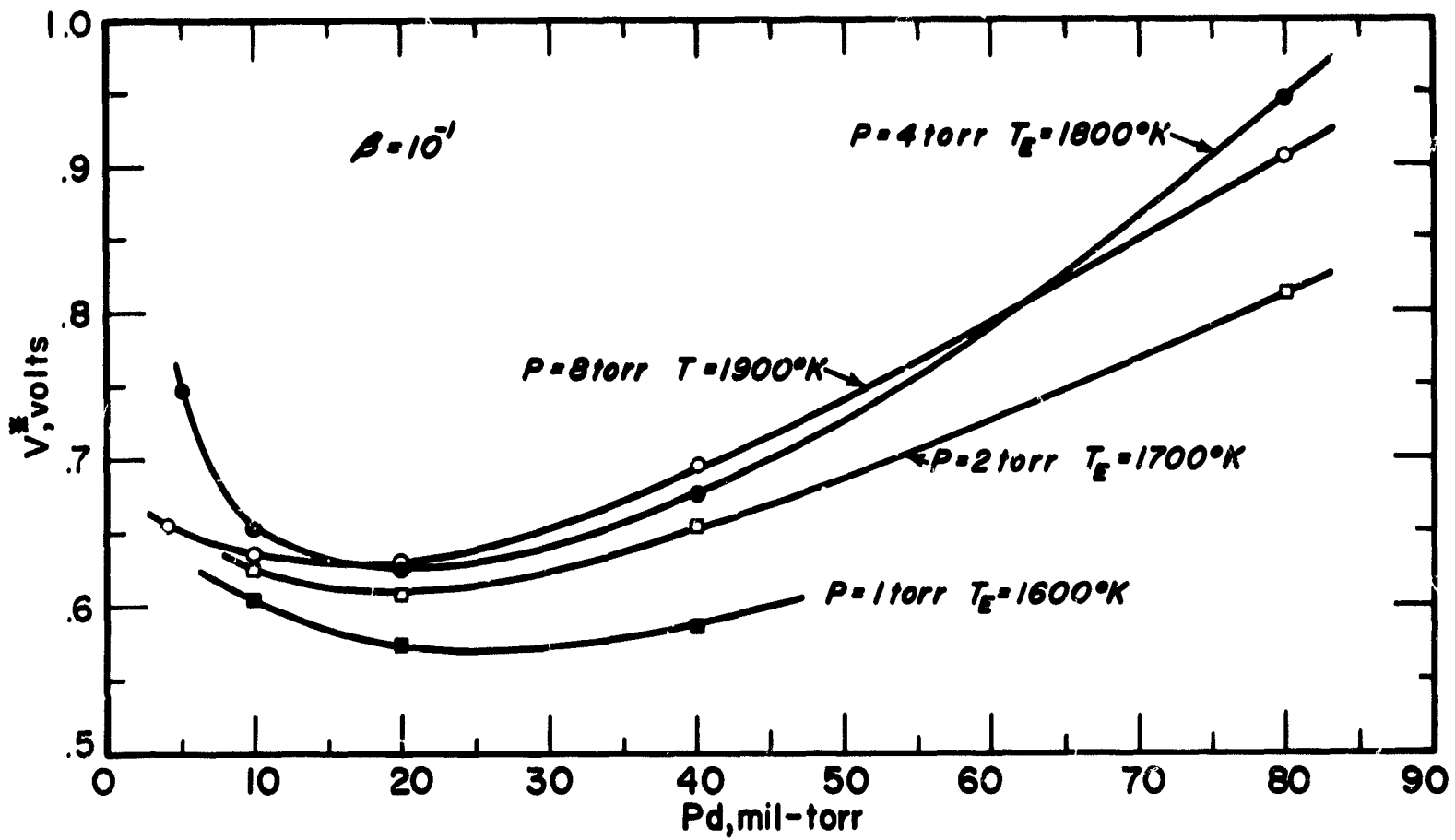


Figure III-49. Voltage Loss Correlation for Several Cesium Pressures at $\beta = 10^{-1}$.

68-TR-10-54

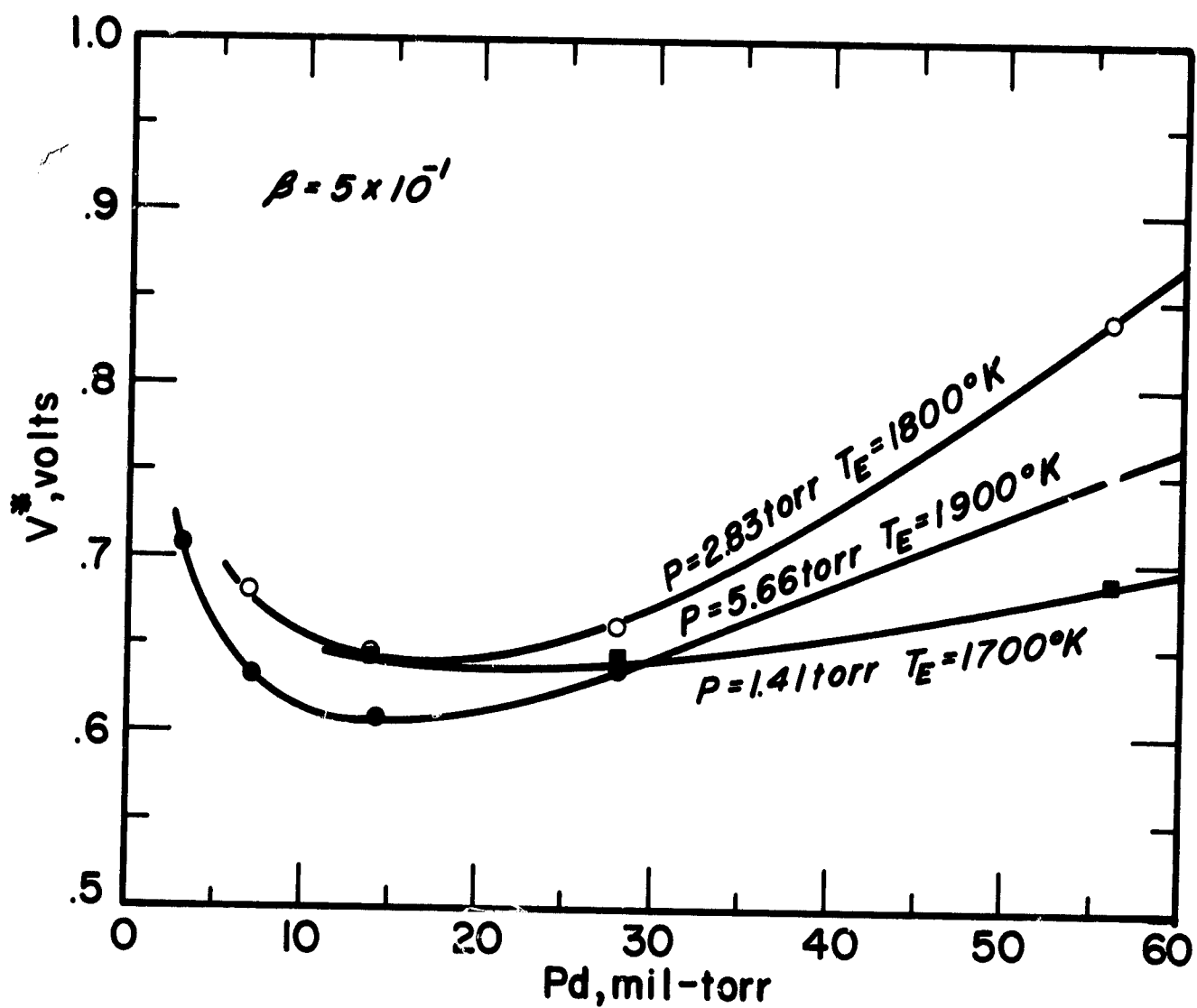


Figure III-50. Voltage Loss Correlation for Several Cesium Pressures at $\beta = 5 \times 10^{-1}$.

68-TR-10-53

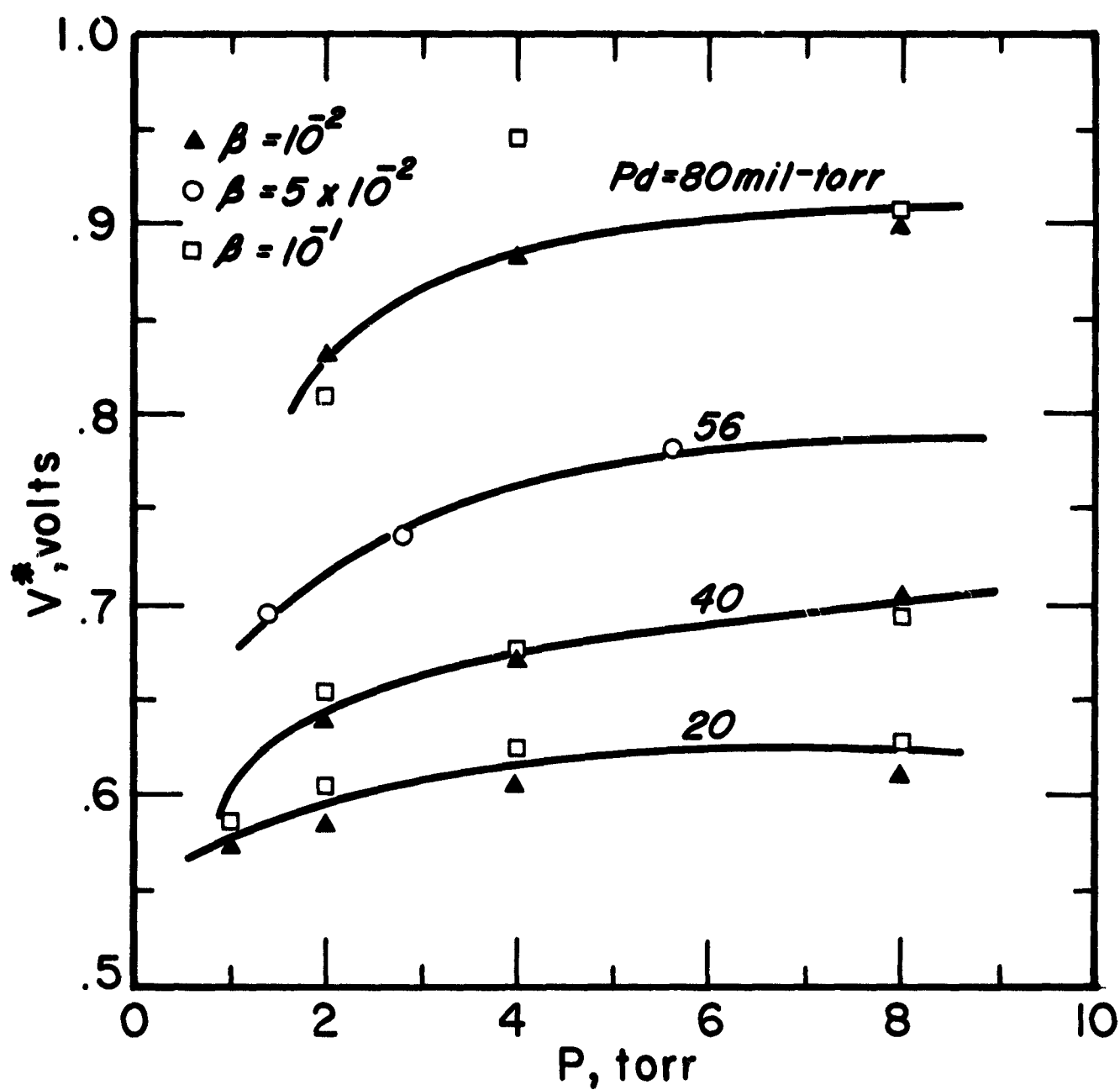


Figure III- 51. Dependence of V^* on Cesium Pressure and Ion Richness.

68-TR-5.4

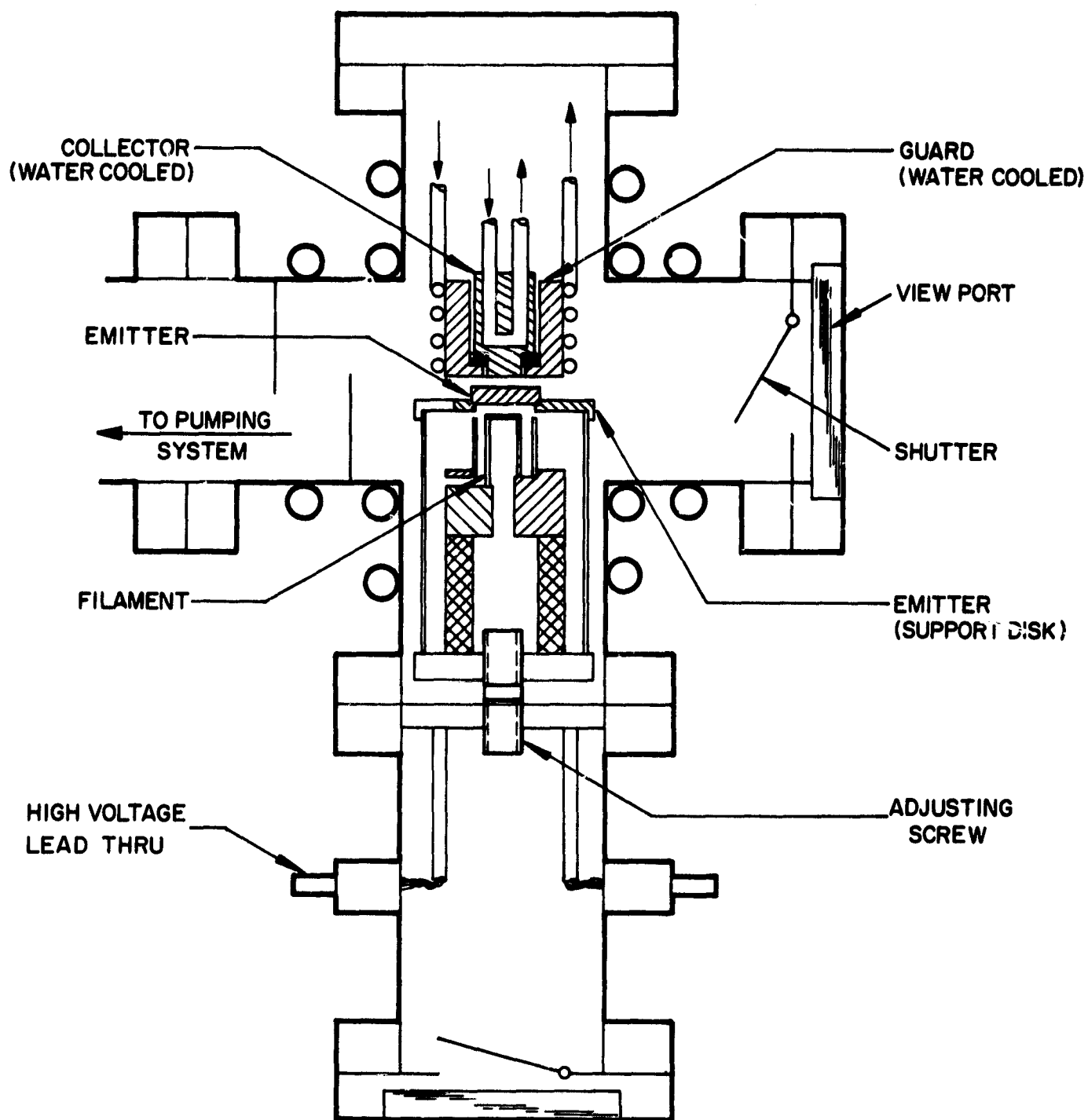


Figure III-52. Apparatus for Vacuum Work Function Measurement.

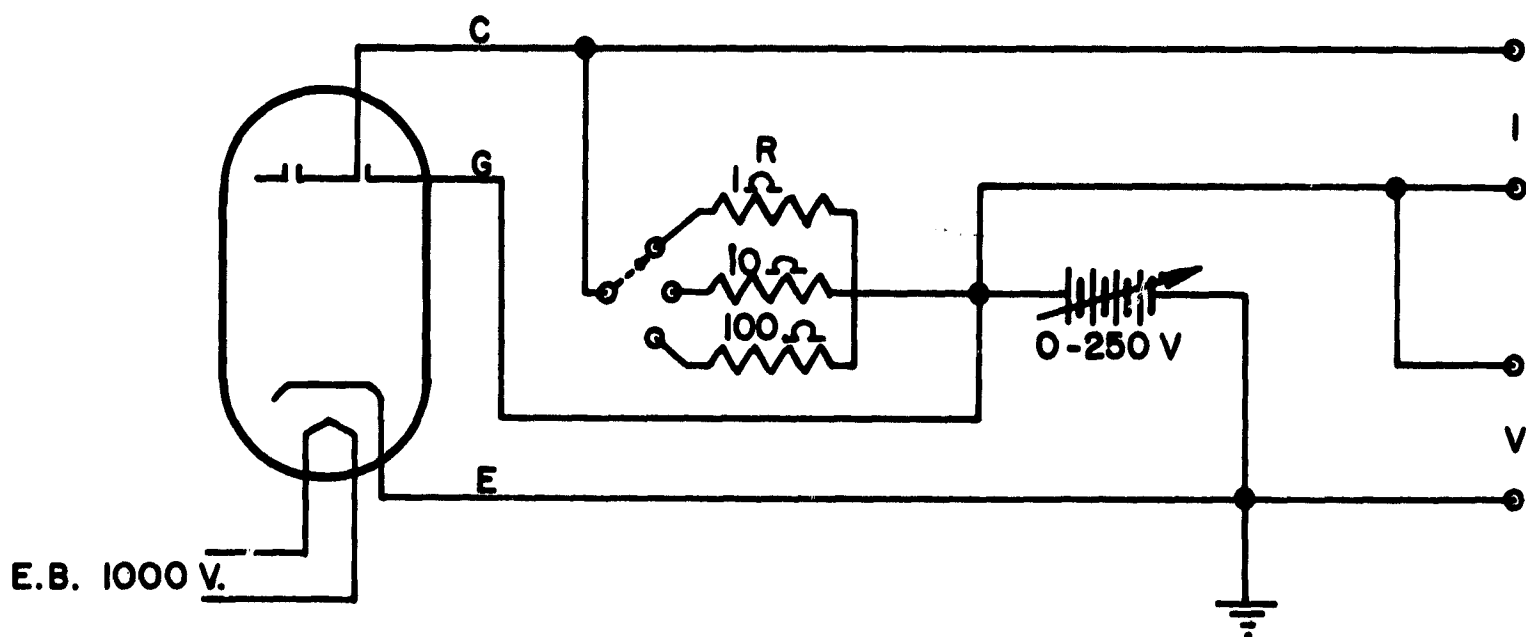


Figure III-53. Circuit Diagram for Bare Work Function Measurement.



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68-TR-11-1

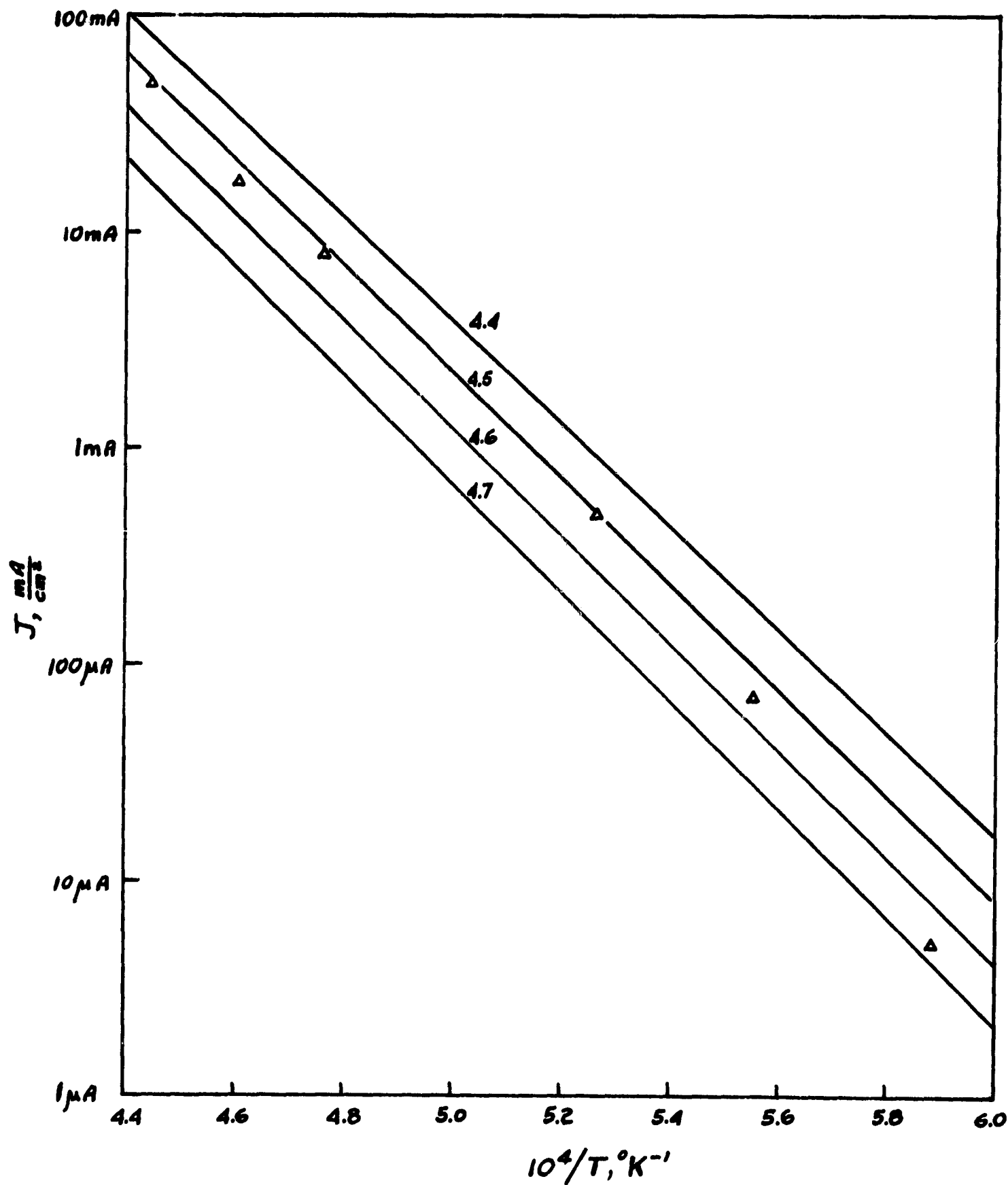


Figure III-54. Bare Work Function Data for Fluoride
Emitter WF-3.