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~~THE~~ PERKIN-ELMER CORPORATION
AEROSPACE DIVISION
2855 Metropolitan Place, Pomona, California 91767

FINAL REPORT
PLANETARY ATMOSPHERIC
EXPERIMENTAL TEST
MASS SPECTROMETER SYSTEM

Contract NAS5-11185
SPO Number 30001

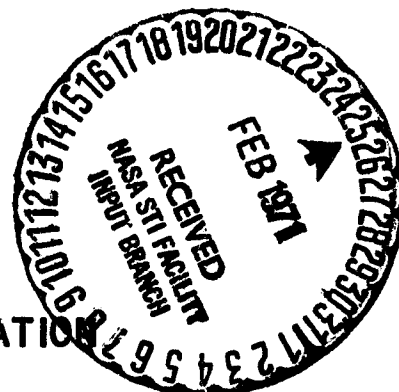
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Goddard Space Flight Center
Greenbelt, Maryland



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MASS SPECTROMETER SYSTEM

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SUMMARY

This is a final report of the design, fabrication and testing of four ion sources and three quadrupole rod and multiplier assemblies. Three of the ion sources are of the dual filament type, two of which were shipped assembled with the quadrupole analyzer. The third ion source was shipped fully assembled and tested, but not attached to the analyzer. The third analyzer was assembled with an open ion source and shipped assembled without the housing.

The three Quadrupole Mass Spectrometer Systems are assembled on a flat plate where all the electrical feedthroughs are located. Also, the ion source, rod and multiplier assemblies are mounted on this baseplate through vibration isolation flexure plates. The two dual filament quadrupole systems were completely welded to the housing before final testing and shipment. The test results were the best ever achieved by a flight quadrupole. This is primarily due to the masking of the low amplitude ions (see the Mask Study Report, Contract NAS5-11185).

As part of this contract, four study efforts were completed. These studies included optimization of a quadrupole system, the effect of masking of ion sources in a quadrupole, the use of segmented rods, and a study of inlet sampling tubes for reentry vehicles. Final reports have been completed in all of the above studies.

The test results along with the studies is believed to have advanced the state-of-the-art of quadrupole mass filters.

INTRODUCTION

The initial contract, NAS5-11185 as defined by the Statement of Work, was for four cylindrical rod quadrupole mass spectrometers reflecting the integration of specialized instrumentation developed and produced under Contract NAS5-11045 and NAS5-3453. During March of 1969 the contract was modified; however, to reduce the quantity from four cylindrical rod systems to three hyperbolic rod systems with extensive modification in the design. The contract also added four new design study activities.

DESIGN

The Mass Spectrometer (MS) was redesigned to provide improved structural stability of its subcomponents, and to reduce the stresses imposed on the subcomponents by the vacuum envelope and the environment to which the MS is exposed. The rod design is composed of four hyperbolic rods with an

$r_0 = 0.200''$. These rods are held together by straps in two ceramic ring plates and the ceramics are in turn held into two flecture plates on either end of the rods. The ion source, of the dual filament type, is also bolted onto the flecture plate (Figure 1) with alignment of the source and the rods being automatically accomplished by the source nozzle and the ceramic plate. The electron multiplier is also mounted in a flecture-type mounting bracket, resulting in a decoupled system of critical components from the housing (Figure 2). Such a "floating" design provides good vibration isolation of the MS from external environments while maintaining alignment of the internal components. The electron multiplier was redesigned in such a manner as to eliminate the neutral particles, passing through the quadrupole filter from being registered as a signal; thus decreasing the background noise. This elimination of neutrals was accomplished by utilizing the first dynode of the multiplier as an ion deflector guiding the ions to the second dynode. Since the neutrals cannot be bent by electric fields they would continue on a straight line impacting the first dynode and be eliminated. The field geometry between the first and second dynodes is such as to eliminate any secondary electrons produced by the neutrals at the first dynode from possible entry into the second dynode area where the multiplication of the electrons begins to occur.

The quadrupole housing consists of two main parts, the baseplate and the cover. The flecture plates are mounted on the baseplate with all the integral components mounted on the two flecture plates plus the multiplier flecture.

This configuration affords complete access for the electrical connections that are connected to electrical feedthroughs located at the baseplate.

The system is tested in this nude configuration inside a test chamber with an inlet tube inserted in the ion source to obtain the differential pumping required.

After the systems were checked in the test chamber, they were removed and the covers were mated and welded in place. The complete unit was then tested to the approved accepted test procedure and delivered to the customer.

After delivery of the first unit, however, the contract was modified to include the open ion source mated and tested with the second rod assembly and multiplier. This system was delivered without the housing cover. The deliverable items were two completely enclosed and welded dual filament-type quadrupole systems, one nude open ion source quadrupole system and one assembled dual filament ion source.

DESIGN STUDY AND DEVELOPMENT PROGRAMS

As part of the contract, four design study activities were added by the March 1969 modification. These activities included both theoretical and experimental work in ion entrance and aperture masking conditions. Theoretical considerations of the use of segmented rods, sampling studies and improved optimization of quadrupole size, weight and power.

The study of the ion entrance aperture mask included extensive computer acquired data with results showing that masking the ions entering the quadrupole very near the X and Y axes does indeed improve the resolution of the system.

The resolution of the quadrupole is largely limited by the tails on either side of the peak. These tails are caused by ions which are theoretically unstable, but possess a small amplitude at the end of a finite length quadrupole. These penetrating unstable ions occur at working points of the scan line just outside the stability region. With their amplitude, of course, being proportional to the distance between a given working point and the stability region. Other parameters, such as resolution (scan line slope), phase of entry and initial angle and amplitude also play a large role in determining the amplitude or size of the tails.

As shown in the mask study, the most penetrating unstables for any working point on the scan line are ions with small initial amplitude and angle. This is true for all phases of entry. There are conditions where a large initial amplitude and large initial angle can make an ion very penetrating for a given quadrupole length, but this is generally restricted to a few entrance phase angles. The conclusion derived from the theoretical and computer analyses is, that elimination of ions with small initial amplitude improves the performance of a quadrupole mass spectrometer. This elimination is accomplished by a cross grid at the exit side of the nozzle.

A tradeoff of improved data quality versus sensitivity occurs, however, when partial elimination of ions occur. In the actual quadrupole system delivered the optimum theoretical tradeoff conditions were obtained when 0.003 by 0.002 inch wires were crossed over the nozzle opening of 0.010 inch diameter.

The dual filament systems delivered had two types of masks installed. One had a 0.020 inch diameter hole with a rectangular mask of 0.009 by 0.013 inch and a 0.003 by 0.002 inch cross etch in the center, while the second had a 0.010 inch diameter aperture with a 0.003 by 0.003 inch wire cross. As expected, the first condition provided somewhat improved sensitivity while the second provided improved quality data (resolution, etc.).

The study of segmented rods indicated that improved data could be obtained by a quadrupole using an ac delayed dc ramp (or segments) at the expense of additional weight and length.

This study shows that the sensitivity may not be improved by a factor of more than two or three using segmented rods versus the sensitivity that may be obtained by correct utilization of quadrupole biasing, using a simultaneous ac and dc ramp.

CHRONOLOGICAL EVENTS AND TEST RESULTS

The dual filament ion source was first installed in the analyzer on 9 June 1969. The instrument operated at a lower quality level than the operation obtained with the high pressure source coupled with the flight analyzer. Figures 3 and 4 show the data obtained under the above conditions.

Considerable effort was expended to optimize the operation of the dual filament ion source. This effort was directed towards obtaining a set of operational curves with varying conditions so that the optimal operating parameters could be determined. Some of these operational curves are shown in Figures 5 through 8. The best data was obtained with reduced ion energy and ion energy spread, as shown in Figure 9. When Figure 9 (best dual filament source data) and Figure 3 (high pressure source data) are compared note that the high pressure ion source provided superior data to that of the dual filament source. There was a considerable sacrifice of intensity to obtain the data shown in Figure 9. At this time, the Aperture Mask Study was just beginning and the decision was made to try a Y-axis mask by placing an 0.001 inch wire in the appropriate orientation at the nozzle. Several scans were made, but there was no appreciable change of the peak shape except for the reduction of the intensity because of the wire masking some of the ions.

The rod polarity was reversed to determine the effect in the X-axis, but again there was no appreciable change in data. The reason for the lack of improvement became obvious after the mask study was completed, but at the early stages of the investigation there were no guide rules to follow to indicate the size and effect of the mask. Later, when the masking effect was better understood it was calculated that an 0.001 inch wire would only reduce the tails by less than ten percent of their initial value. This is a reduction which could easily go unnoticed with all the other variations that can occur.

As mentioned above, having no guide rules to go by at that time, it was decided to try an upper limit of wire size to make the effect very apparent.

The size of the upper limit was partially determined by the amount of intensity loss which was to be sustained. The wire size was determined to be 0.005 inch. The wire was spot welded onto the nozzle and a scan was run. Figure 10 shows this scan, which obviates the tail reduction when compared with a similar scan (Figure 11) without any mask. The rod polarity was again reversed to determine the tail reduction in the X-axis and again a dramatic decrease appeared, as shown in Figure 12.

A mask of wire size of 0.005 inch with 0.010 inch diameter nozzle, however, would eliminate most of the intensity; thus, the mask was changed to 0.003 inch wire cross which reduced the intensity to about 40 percent of its initial value. A scan of mass 28 region with the 0.003 inch cross is shown in Figure 13. In the same figure, the tail magnitude, as it would appear without a mask, is drawn approximately in the Y-axis.

Considerable effort was spent in testing and optimizing the first system. Every new test conducted with this system appeared to produce excellent data, much improved over any other known quadrupole data. Figure 14 shows a typical linear scan of resolution versus tail slope, while Figures 15 through 19 show a scan over mass 1 to 140. Note that the dynamic range of this system is over six decades.

The operation of this system is so good that resolution of CO_2^+ ($M = 44.0039$) and C_3H_8^+ ($M = 44.0776$) was possible, as shown in Figure 20. This separation requires a resolving power of over 605. Resolving powers of about 900 were obtained with this quadrupole.

The sensitivity of this quadrupole configuration was measured at about 1×10^{-7} amperes per torr. In order to increase the sensitivity, the second system was redesigned to increase the nozzle opening. The aperture was increased to 0.020 inch diameter and a roughly rectangular etched mask was placed at the nozzle exit. The mask has a geometry approximating a cross of 0.003 by 0.002 inch. This redesigned nozzle system was tested and found to possess a sensitivity of about 3.5×10^{-7} amperes per torr. This increase in sensitivity was, however, obtained at the expense of other parameters such as resolution, for example, the 100 percent transmission cutoff point for the first system is about mass 90, while that for the second system was about mass 60.

The second unit was functionally checked in the test chamber. The unit was removed and its cover was electron beam welded and underwent acceptance testing in December of 1969 with a typical scan shown in Figure 21. The unit was hand carried by GSFC personnel on 17 December 1969.

The ion source from the first unit was removed and replaced by the open ion source. Considerable testing was done with this unit in an attempt to optimize the mode of operation. The nozzle of the open ion source is of the same geometry as the second unit, optimized for sensitivity and not for resolution. The sensitivity of the open ion source system was measured at approximately 1.7×10^{-7} amperes per torr. Typical of the optimization efforts is Figure 22, showing the variation of spectrum shape as a function of quadrupole biasing with respect to the accelerator. Figure 23 is a scan that was optimized over the entire mass spectrum shown.

The open ion source system was hand carried to GSFC by Goddard personnel in February 1970. This unit was shipped nude (without the cover).

After the delivery of serial number 003 (open ion source unit) work was stopped on the PAET instruments and design of a proposed four inch rod quadrupole was negotiated. An energetic particle focusing magnetic ion source was also designed at this time. The final design of the four inch quadrupole has a smaller block dual filament ion source with the inlet through one of the anodes. A chamber was also designed as part of the inlet system to thermalize the molecules by wall collisions. The dual filament ion source, in addition to the changes mentioned above, was redesigned so that its conductance was decreased from approximately 50 to about 15 cubic centimeters per second. The hyperbolic rods, along with the decrease in length from six inches to four inches, had the r_0 decreased from 0.200 to 0.100 inch.

The multiplier geometry is also decreased until the entire system, except for the thermalizing chamber, fits inside a cylindrical housing of about 1.75 inches outside diameter by about 10 inches long. All the electrical connections are routed through feedthroughs on either end of the cylinder. The multiplier is a deflector type, as in the above tested PAET instrument, with of course, a smaller geometry. The magnetic ion source is half the size of the open ion source mentioned above.

After the design of the four inch quadrupole was defined, testing of the third PAET instrument was again initiated. This third instrument, which is identified as serial number 001, consists of the dual filament ion source, which was tested from June to October of 1969, with the data shown in Figures 4 through 20 and a third set of rods and multiplier. Serial number 001 has the small nozzle aperture (0.010 inch) with the 0.003 inch wire cross as a mask.

Testing of serial number 001 was conducted in June of 1970 and after acceptance testing was hand carried to GSFC.

Most of the backup optimization data is enclosed in the Acceptance Test Procedure data package. A typical background spectrum is given in Figure 24. A spectrum of air plus background is shown in Figure 25 with the data over mass 28 peak showing the amplitude change with change of high voltage in the electron multiplier. Figure 26 shows the same spectrum with the multiplier high voltage at -2900 volts. Figures 27 through 32 show the variation of spectrum shape with varying operating parameters. All the data shown in Figures 24 through 32 were taken with the unit in the test chamber. After the unit was welded to the cover the acceptance test was run and the data, as usual, improved somewhat because of the more efficient differential pumping. Figure 33 shows the last run taken of the final welded configuration.

At the same time that serial number 001 was delivered the third dual filament ion source was also delivered fully assembled and having a nozzle geometry identical to that of unit serial number 002.

CONCLUSIONS

The Quadrupole Mass Spectrometers developed under this contract have attained the highest operating performance of any known quadrupole of similar size and design.

Empirical data has shown that ion masking improves the performance of a quadrupole mass filter as predicted by theory.

The unique design of an isolation mounted system on a baseplate proved very convenient in assembling and testing of the mass spectrometer system. However, it forced the housing envelope to take the shape of a somewhat unstable (under vacuum) configuration. This in turn necessitated a thicker housing which increased the final weight of the system. The modular assembly developed in this contract has been applied, with modifications, to the four in. rod quadrupole system with apparent success.

The studies conducted under this contract have provided increased knowledge of the operation of the quadrupole system, therefore, it appears that further improvements may be made in the performance of this type of system.

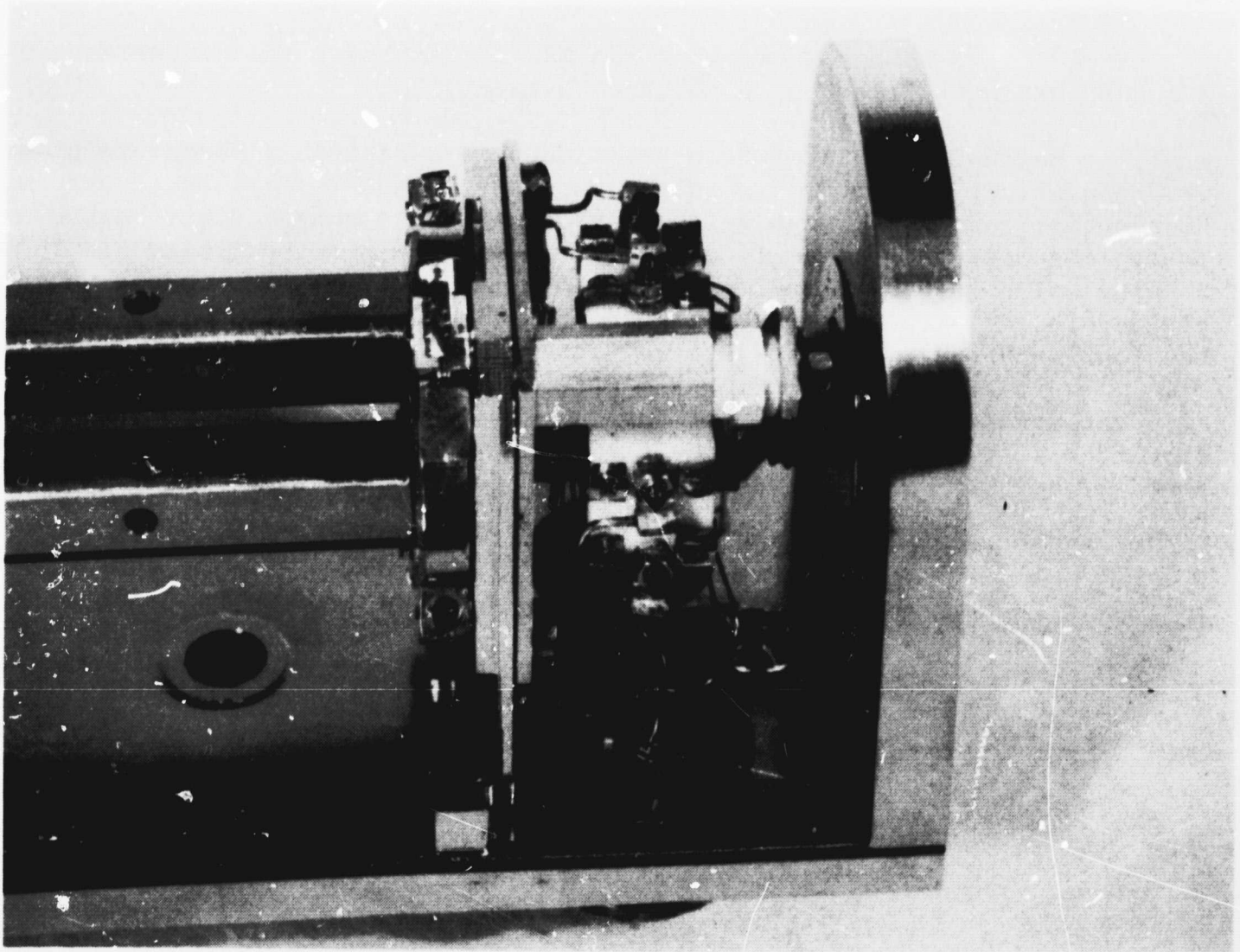


FIGURE 1. Dual Filament Ion Source

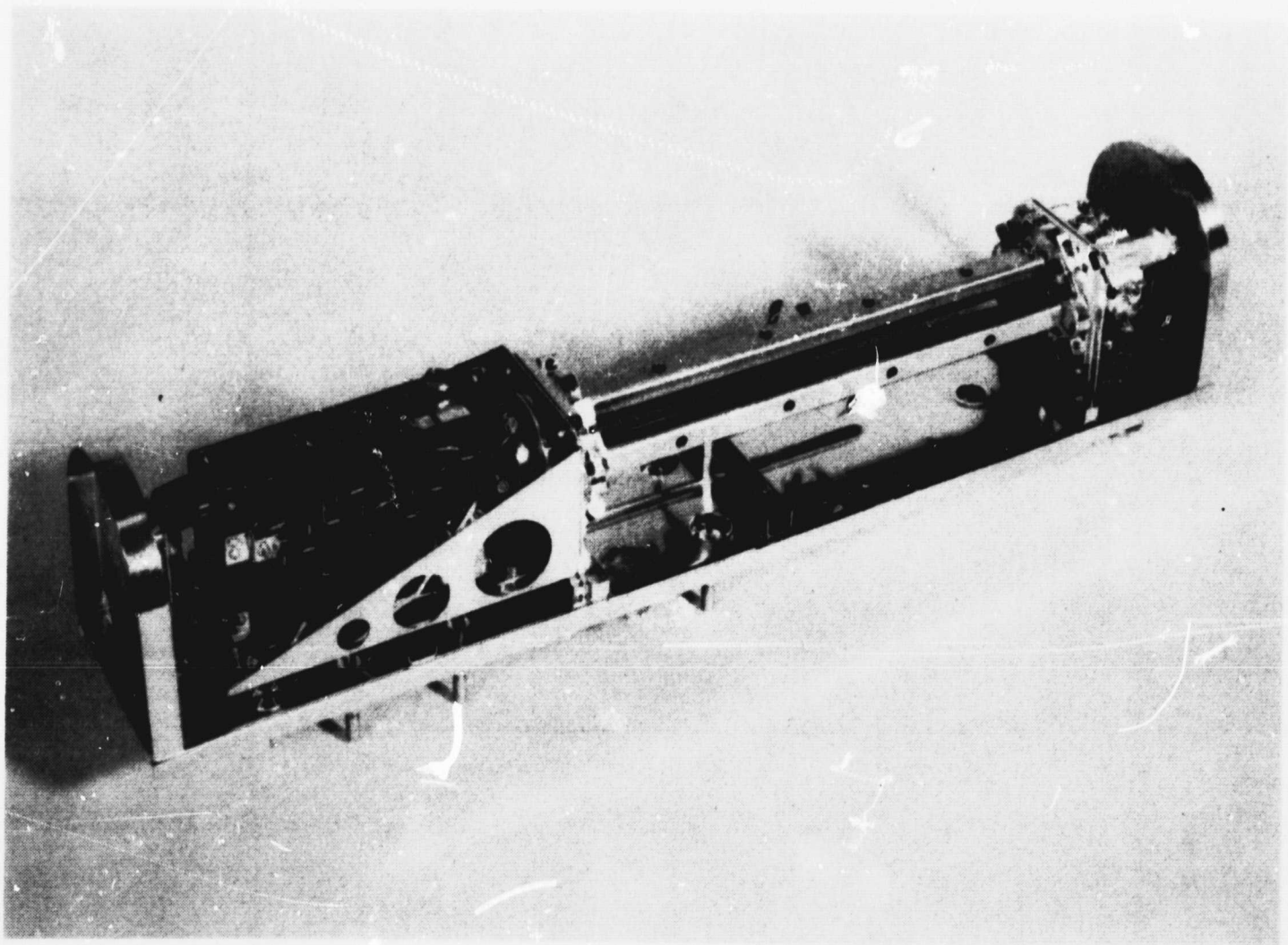


FIGURE 2. Electron Multiplier

RUN #2
IFA 212.5
IFB 208.5
 $R = \frac{77.5}{13.5} \times 7.5 = 43$

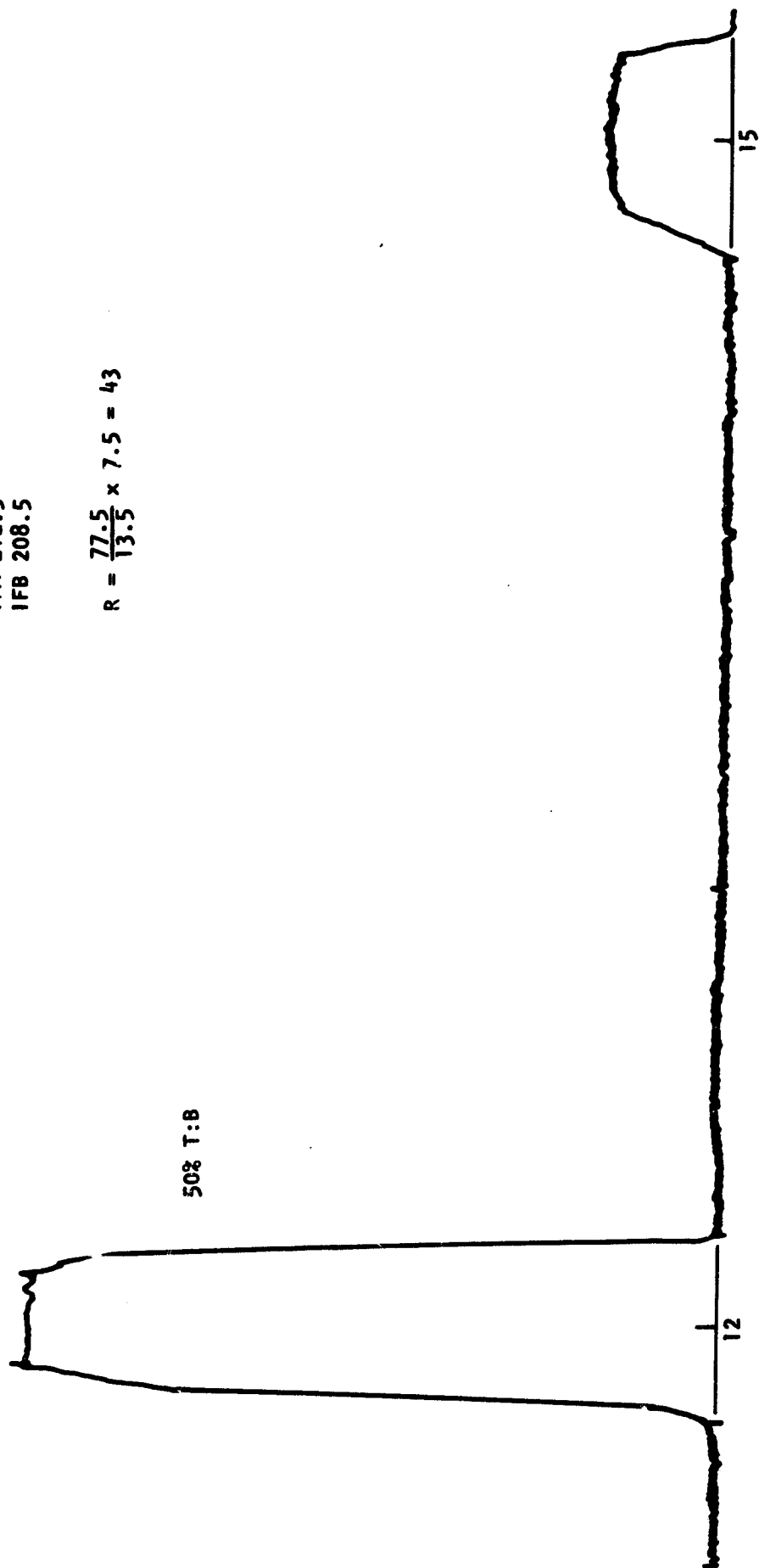


FIGURE 3. High Pressure Ion Source

NOZZLE BIASING TEST

RUN #1

P_{AIR} 1.5 x 10⁻⁶ T

V_{MULT} 2200

IFA 266 V

IFB 0 V

NOZZLE 0 V GND

T:B 45% @ 5%

$$R = \frac{613}{107} \times 7.5 = 43.0$$

$$Q_1 = \frac{1}{430} (1 - 460) = 0.1255$$

$$Q_2 = 0.0735$$

$$S_s = 2.07 \times 10^{-6} \text{ A/T}$$

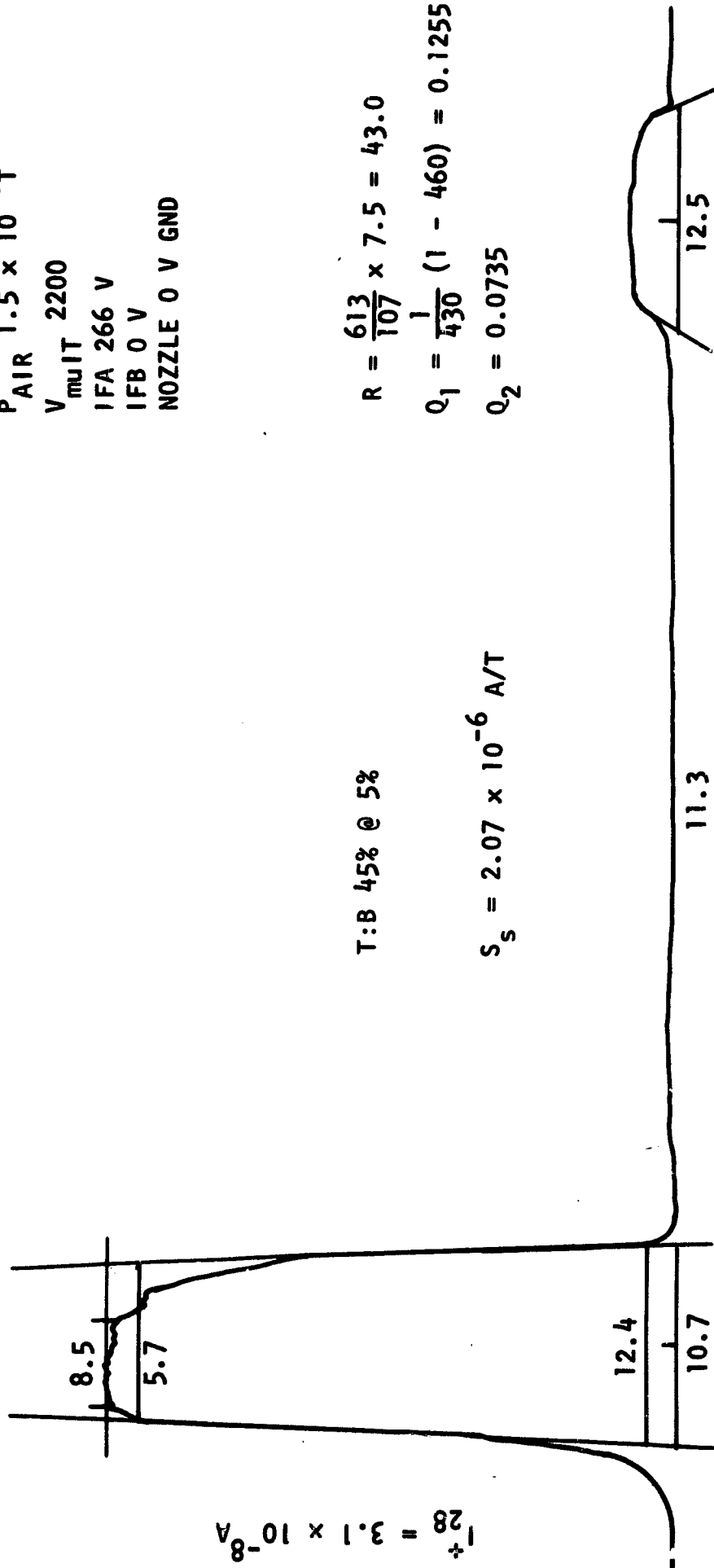


FIGURE 4. Dual Filament Ion Source

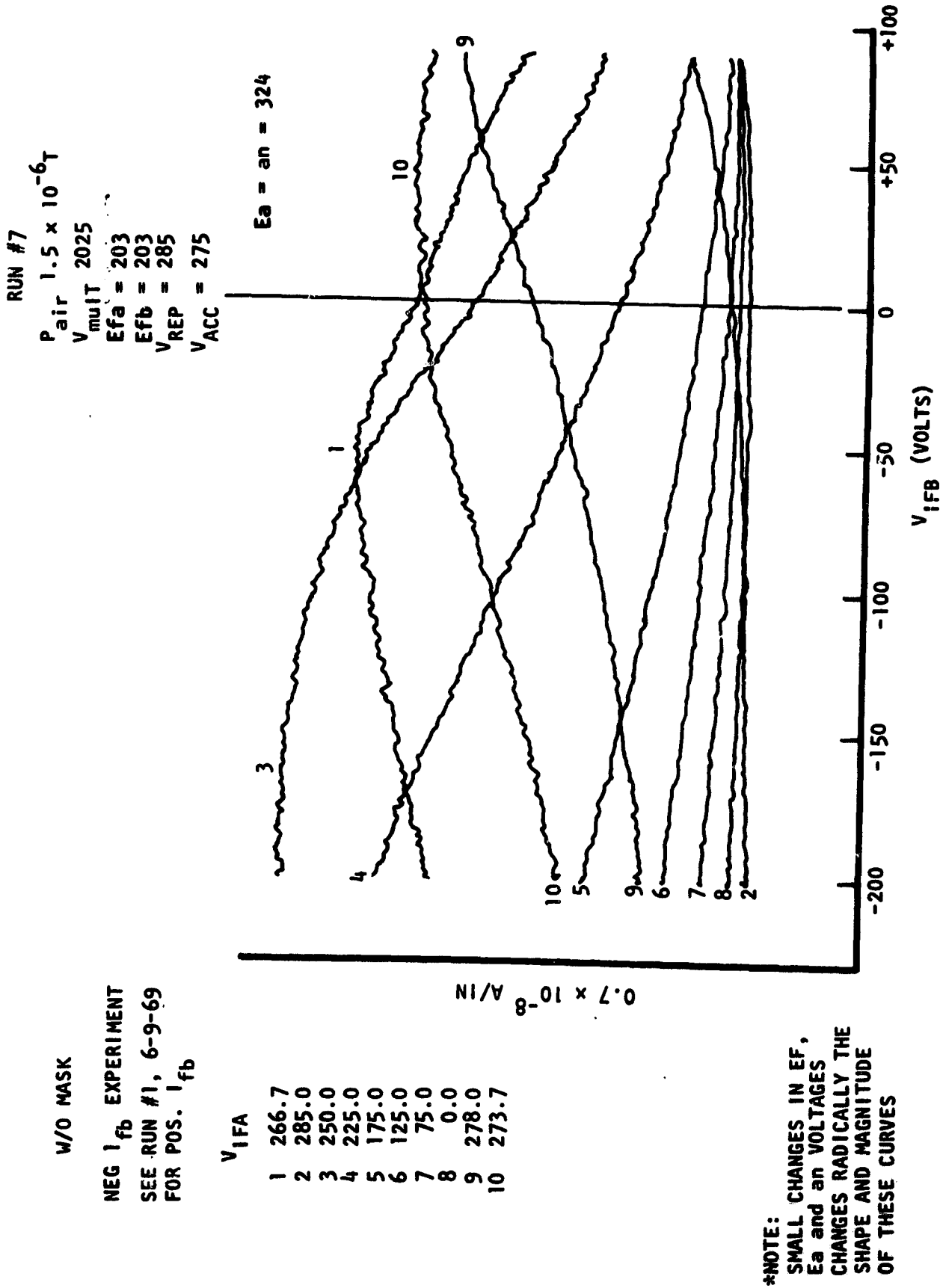


FIGURE 5. Ion Focusing Curves (Lens B)

W/O MASK
 $V_{REP} = 285$
 $V_{ACC} = 275$

	V_{IFA}
1	266.7
2	285.0
3	250.0
4	225.0
5	175.0
6	125.0
7	75.0
8	0.0
9	278.0
10	273.7

RUN #1
 AIR SAMPLE 2.0×10^{-7} P PUMP
 $V_{MULT} = 2025$
 $I_{AN} = 30 \mu A$
 GUN #1
 DUAL FIL SOURCE

$\Sigma a = a_n = 324$
 $\Sigma fa = 203$
 $\Sigma fb = 202$

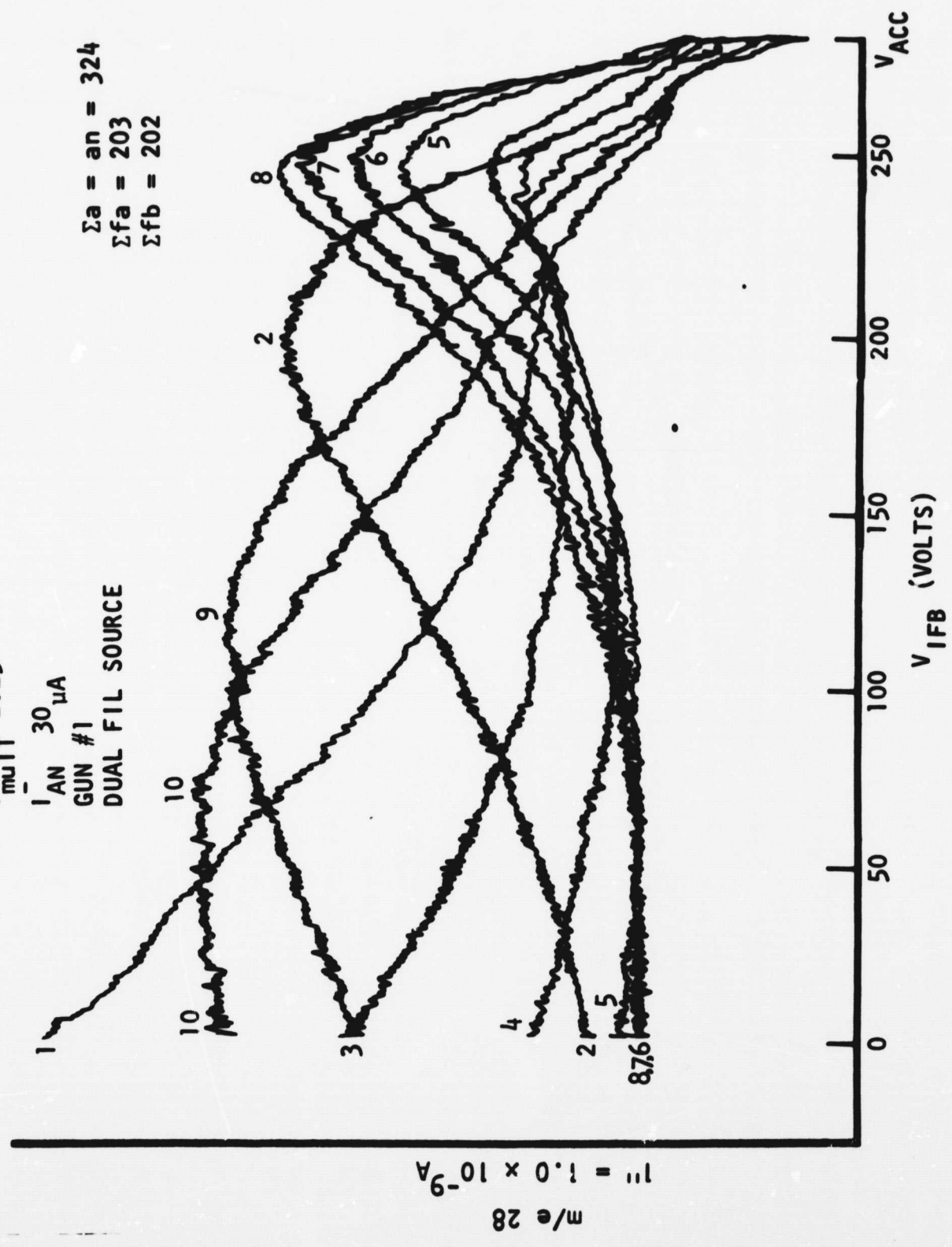


FIGURE 6. Ion Focusing Curves (Lens A)

PEAK SCANED W/QB = 254
 V_{ACC} = 275
 V_{REP} = 285
 V_{NOZZLE} = 0 V
 $V_{el's}$ = 0 V

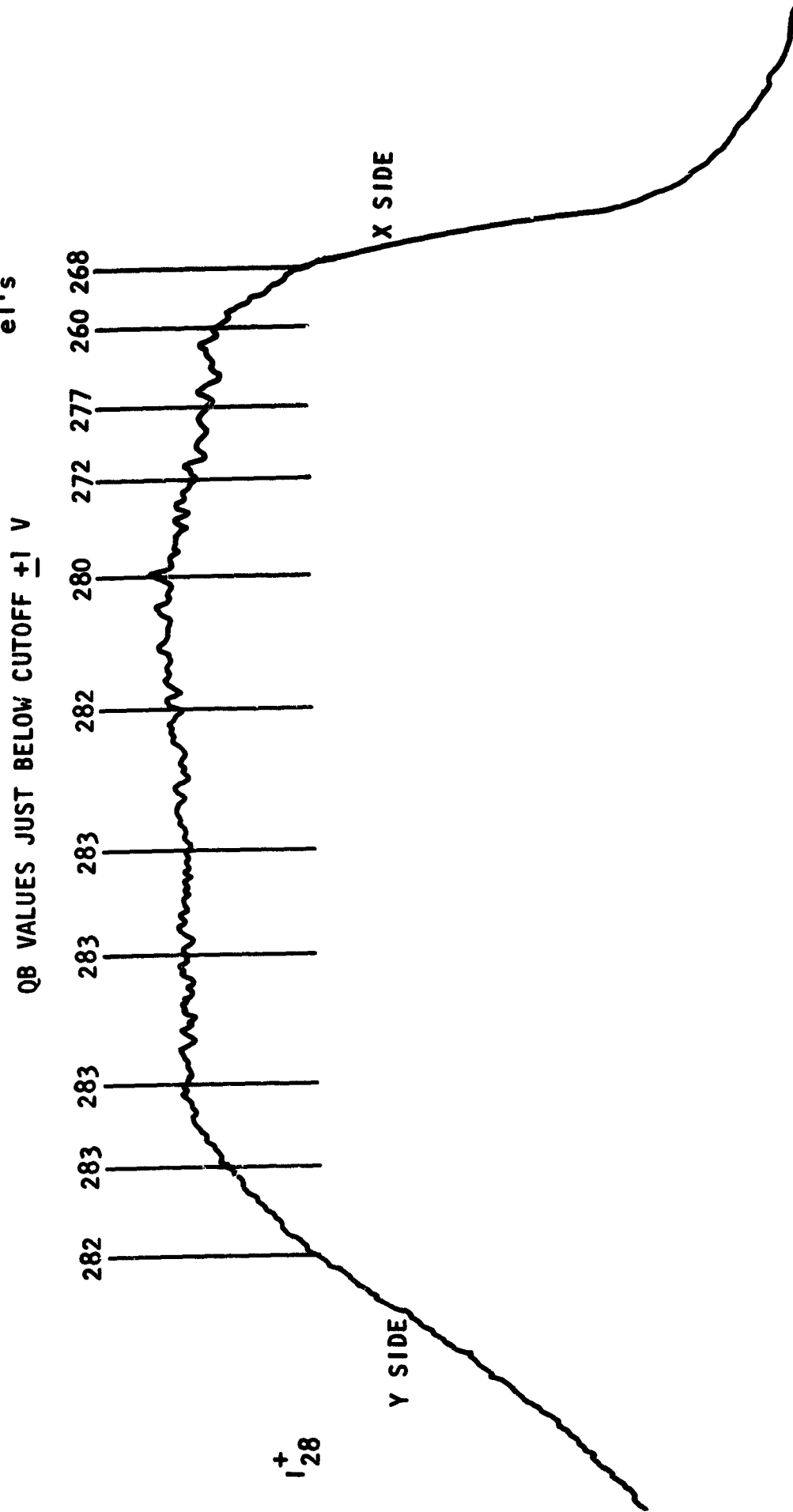


FIGURE 7. QB Cutoff vs Peak Position - Without Mask

$V_{ea} = V_{AN} \quad 1 = V_{ea} \quad 2 = V_{ON} \quad 2 = 324$
 $V_{REP} = 285$
 $V_{ACC} = 275$
 $V_{QB} = 280.5$

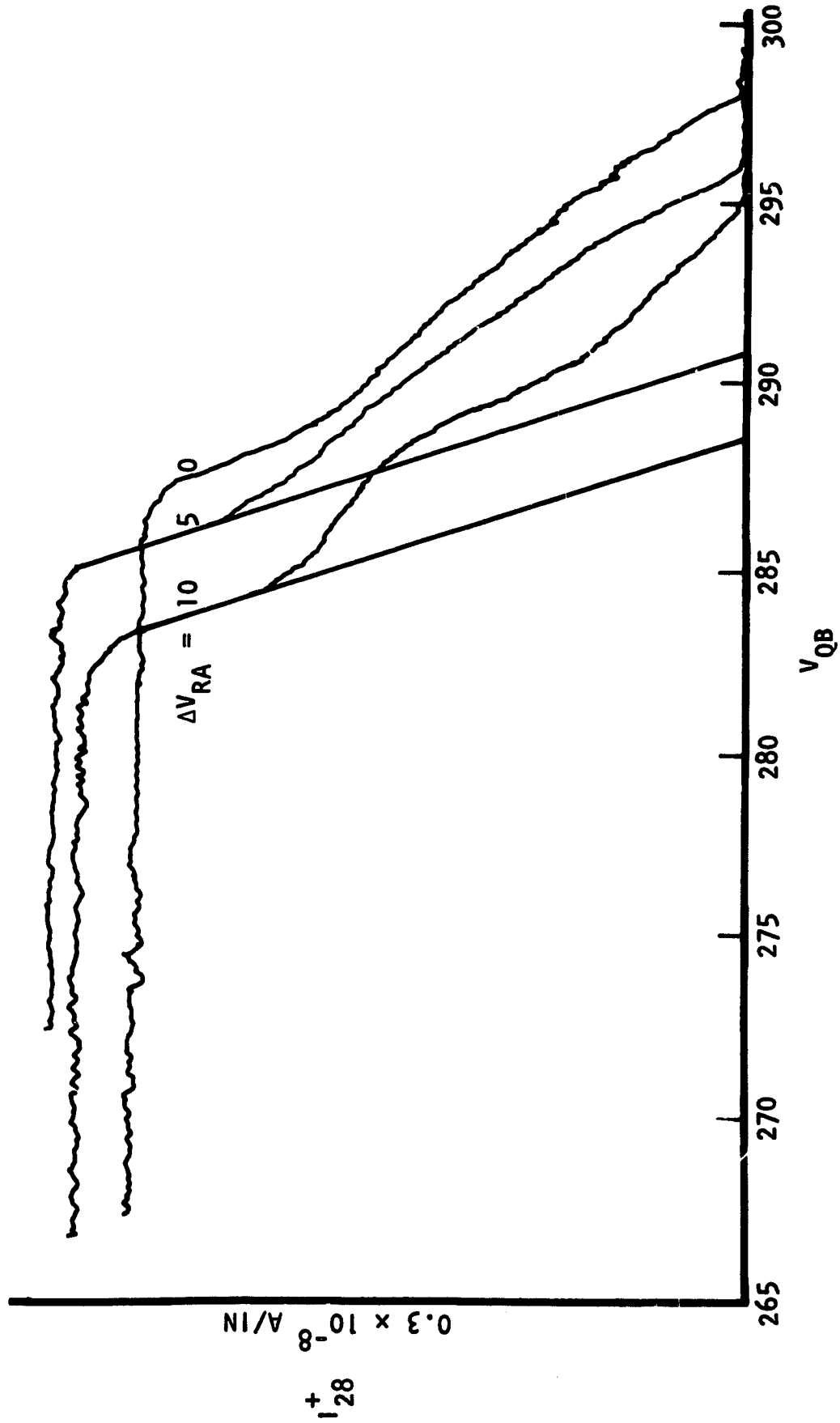


FIGURE 8. Quad Bias Cutoff Runs

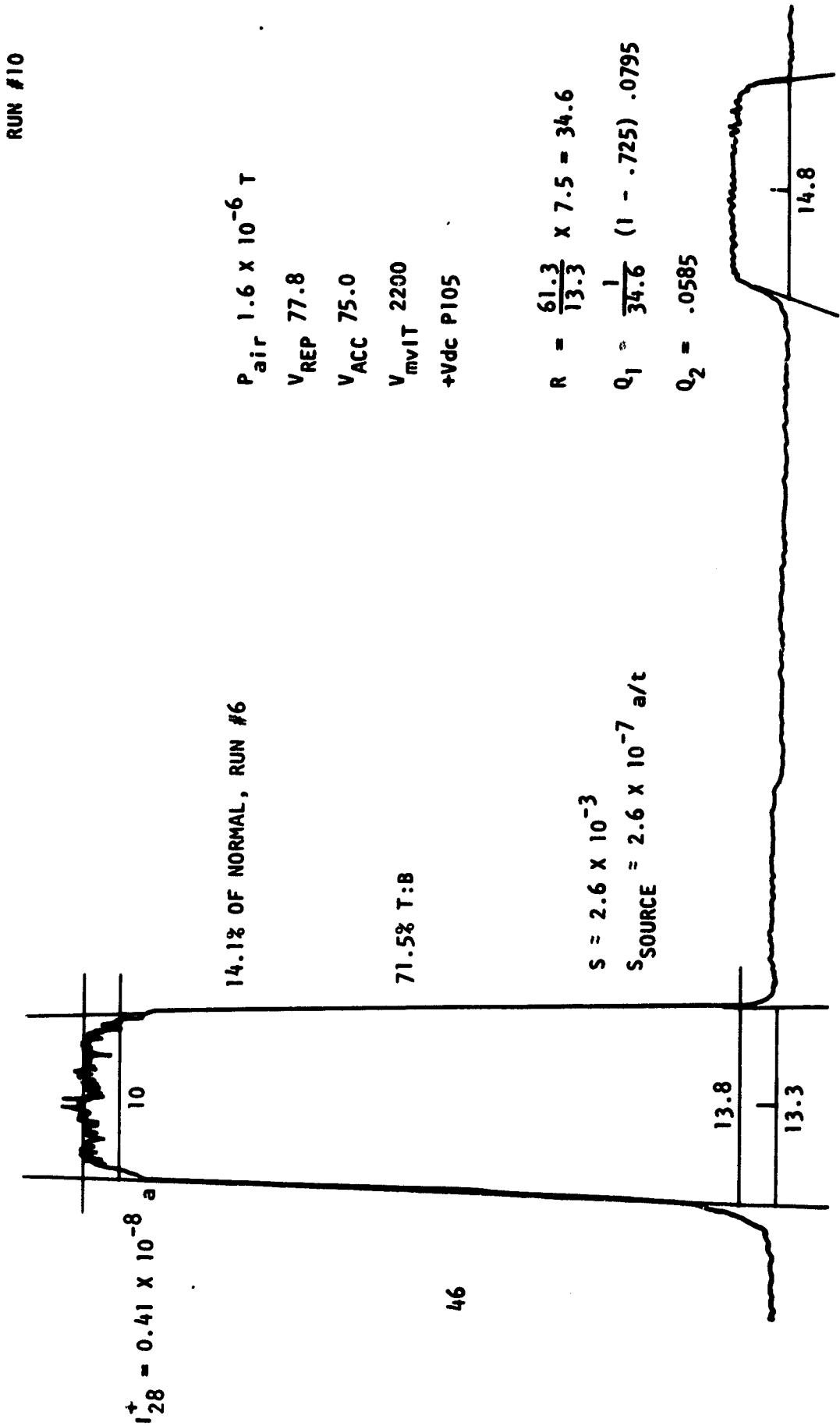
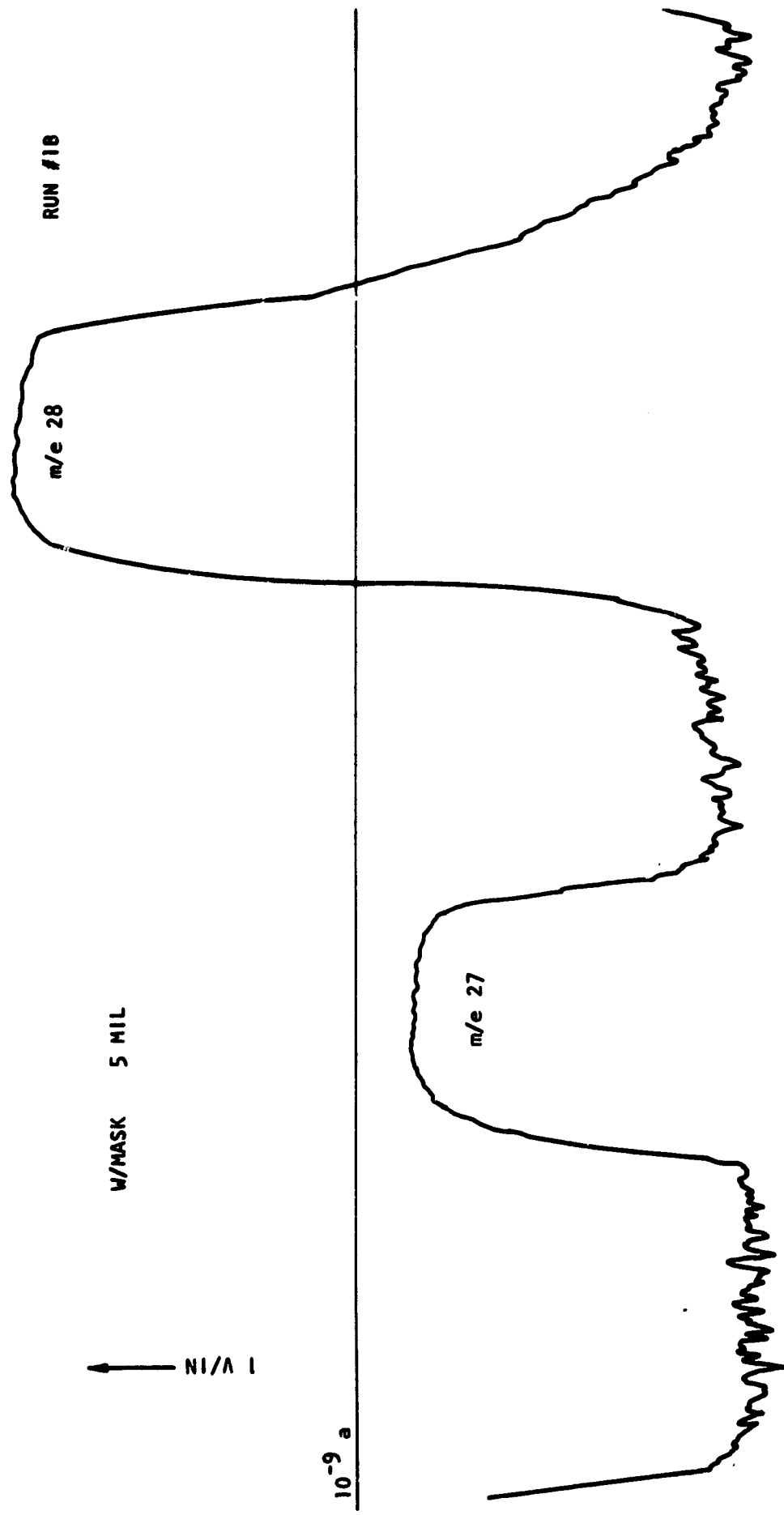


FIGURE 9. Reduced V_{ION} and Proportionally Reduced ΔV_{ION} Test



Rec 0

FIGURE 10. Peak Shape With 0.005 Inch Mask on Y-Axis

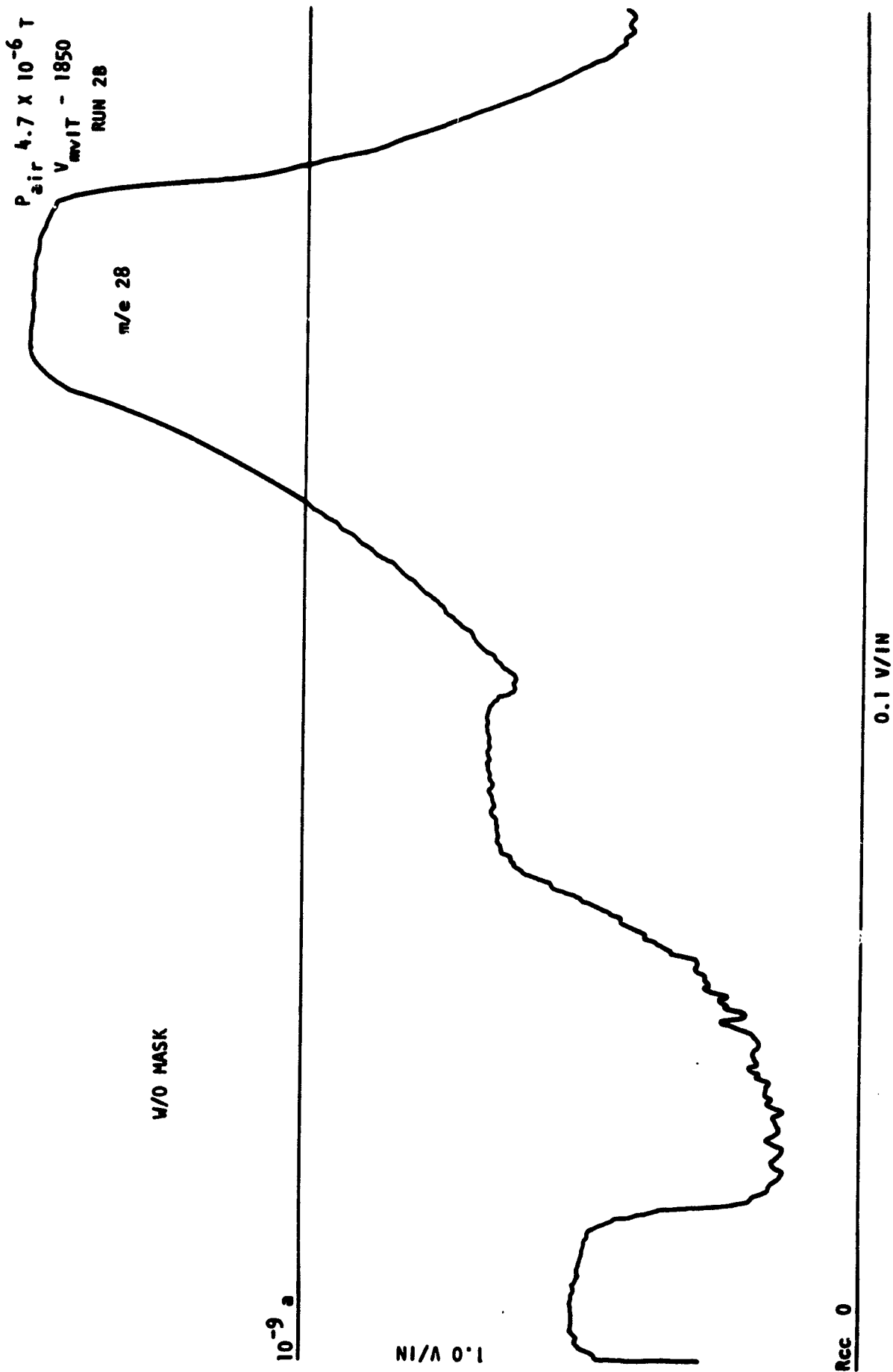


FIGURE 11. Peak Shape Without Mask

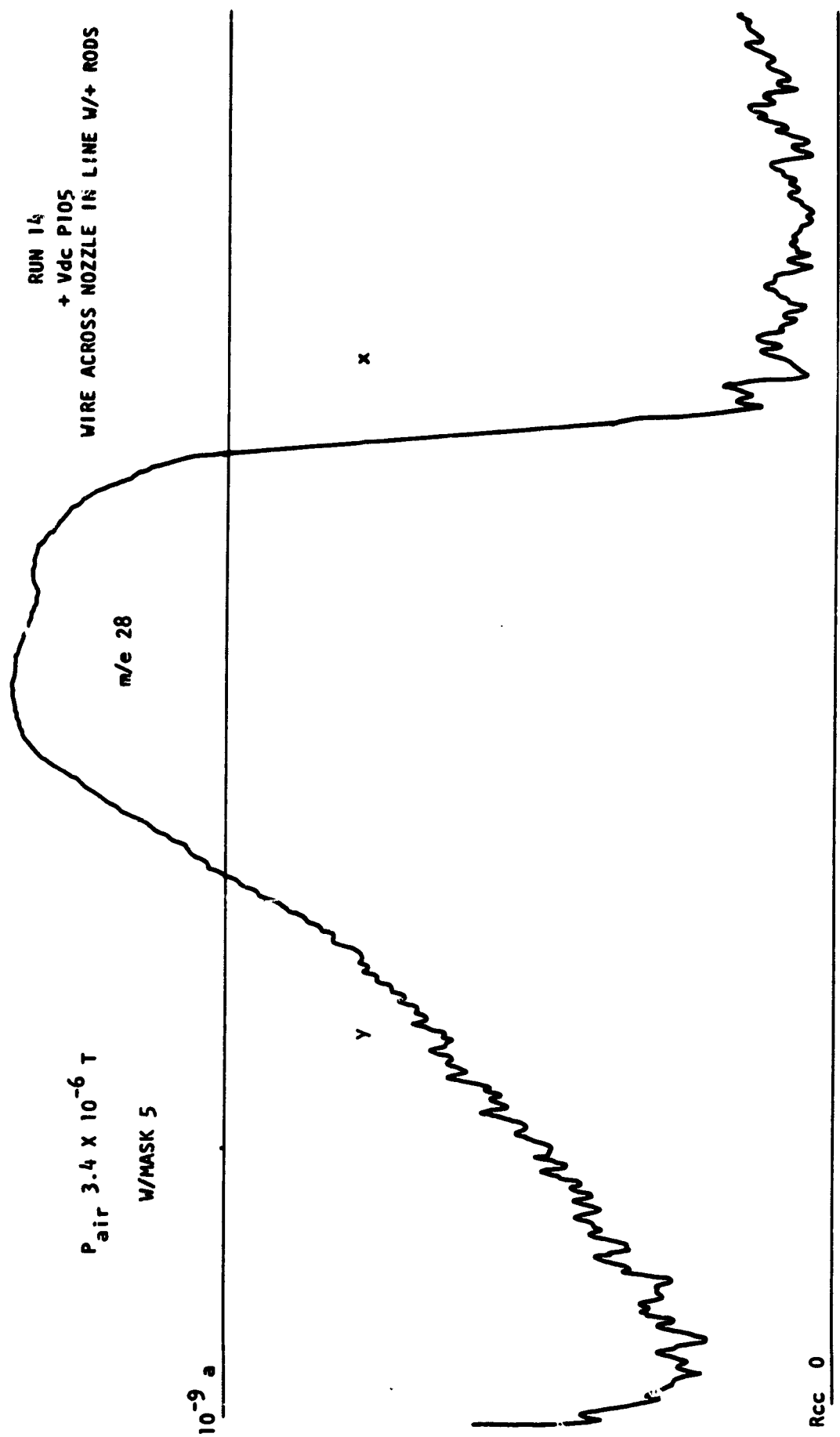


FIGURE 12. Mask on X-Axis

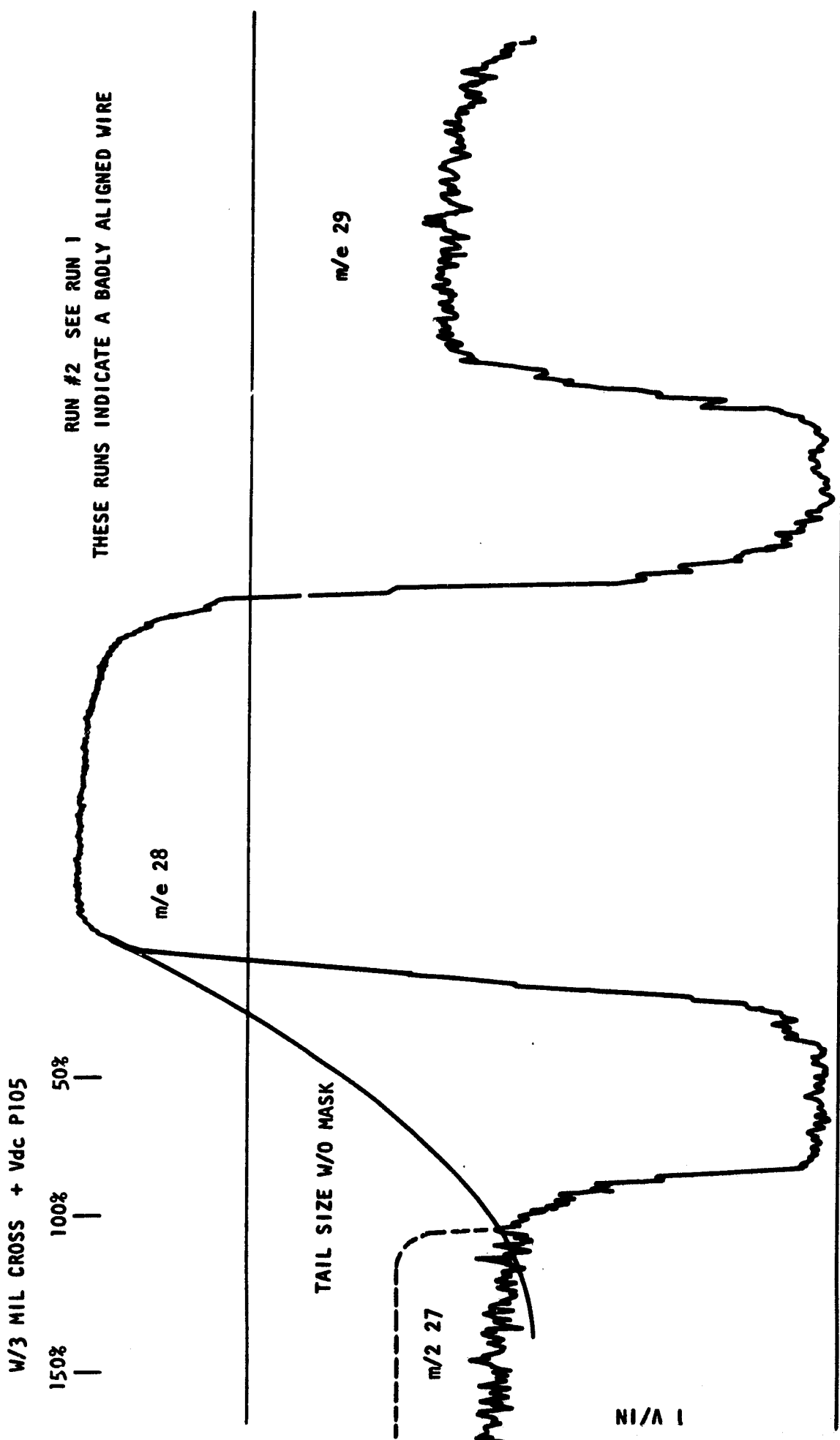


FIGURE 13. Peak Shape With 0.003 Inch Cross (Rod DC Potential Reversed)

W/3 MIL CROSS

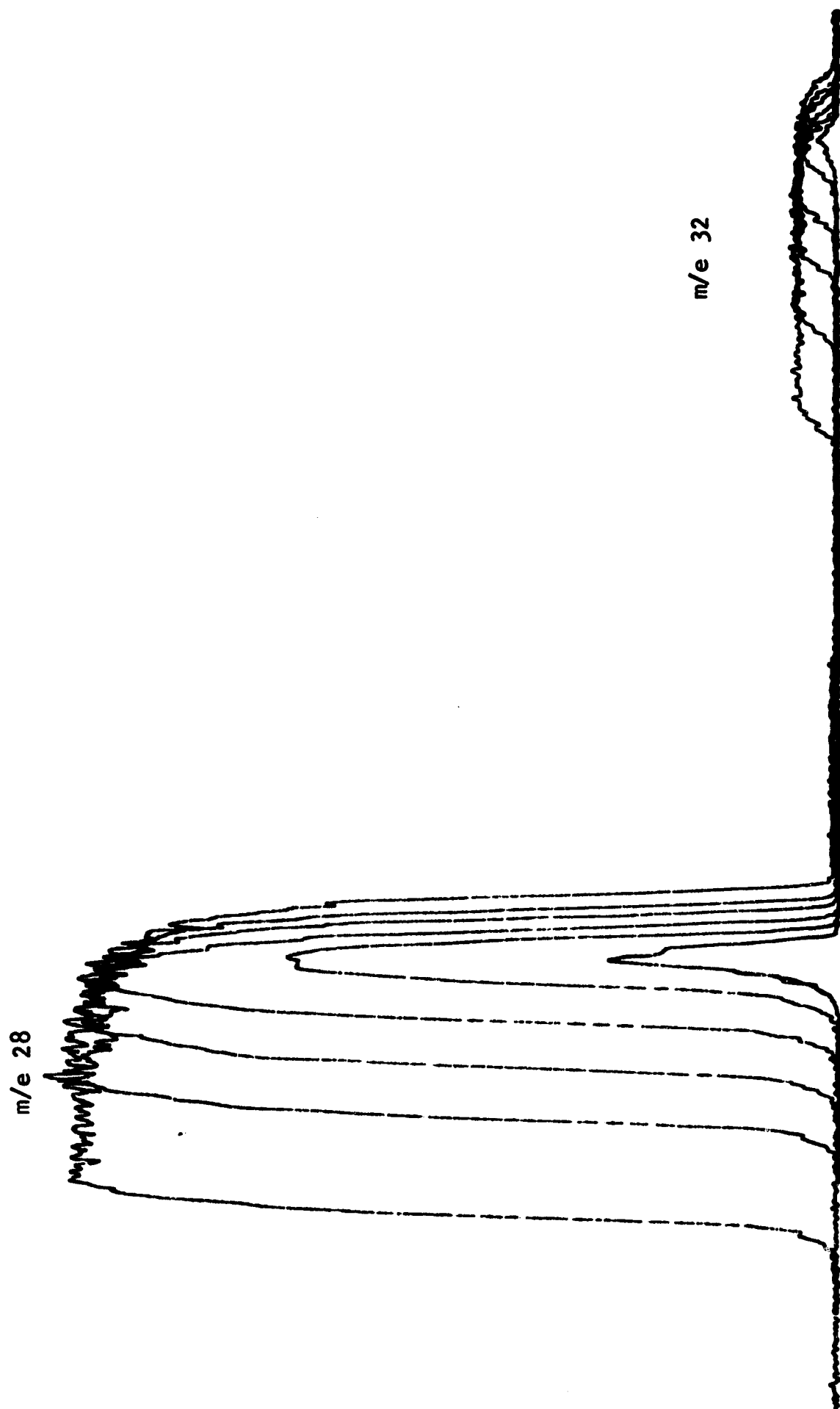


FIGURE 14. Tail Slope Vs. Resolution

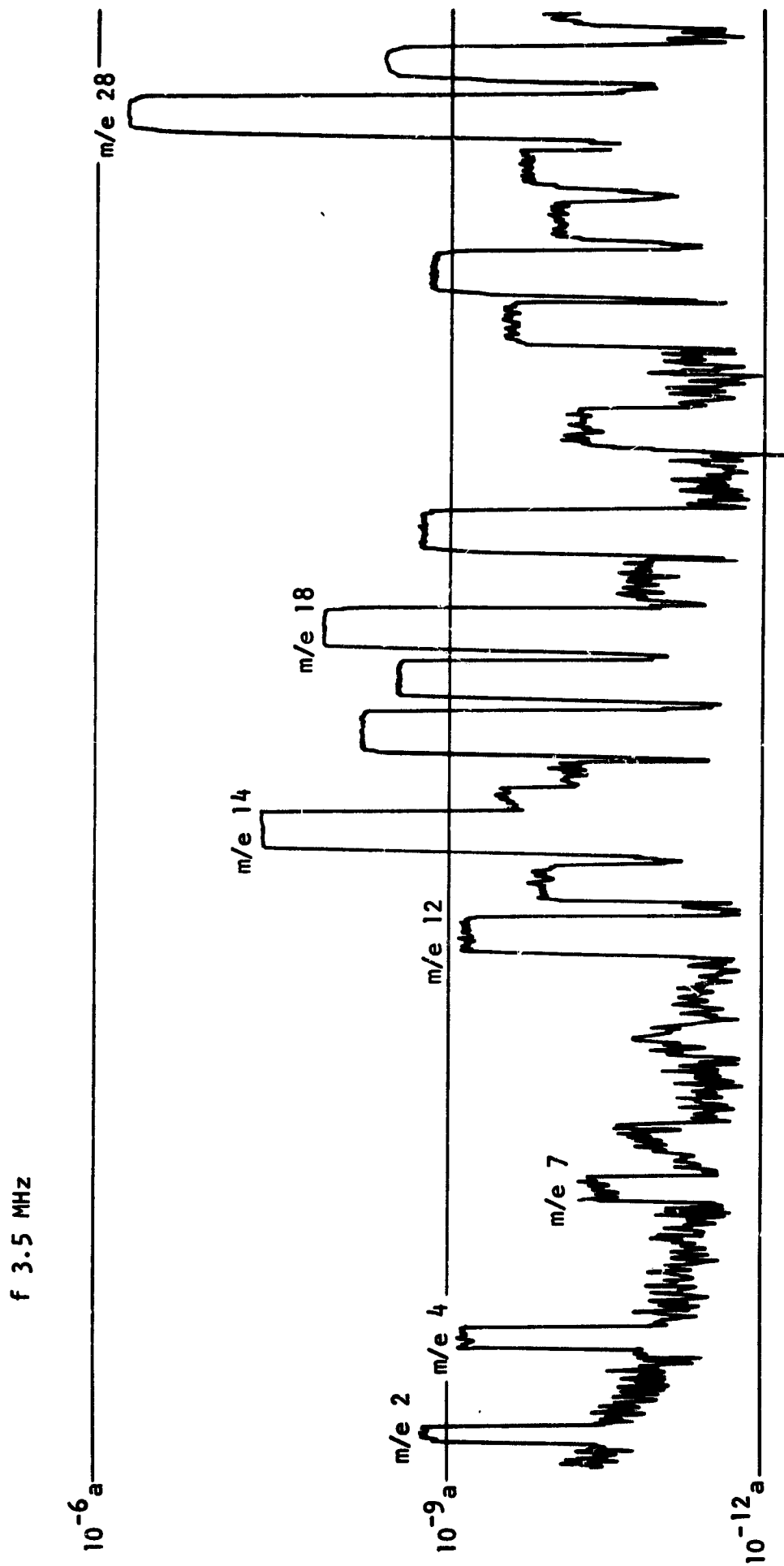


FIGURE 15. Mass Spectrum - m/e 1 to m/e 29

f 214 MHz

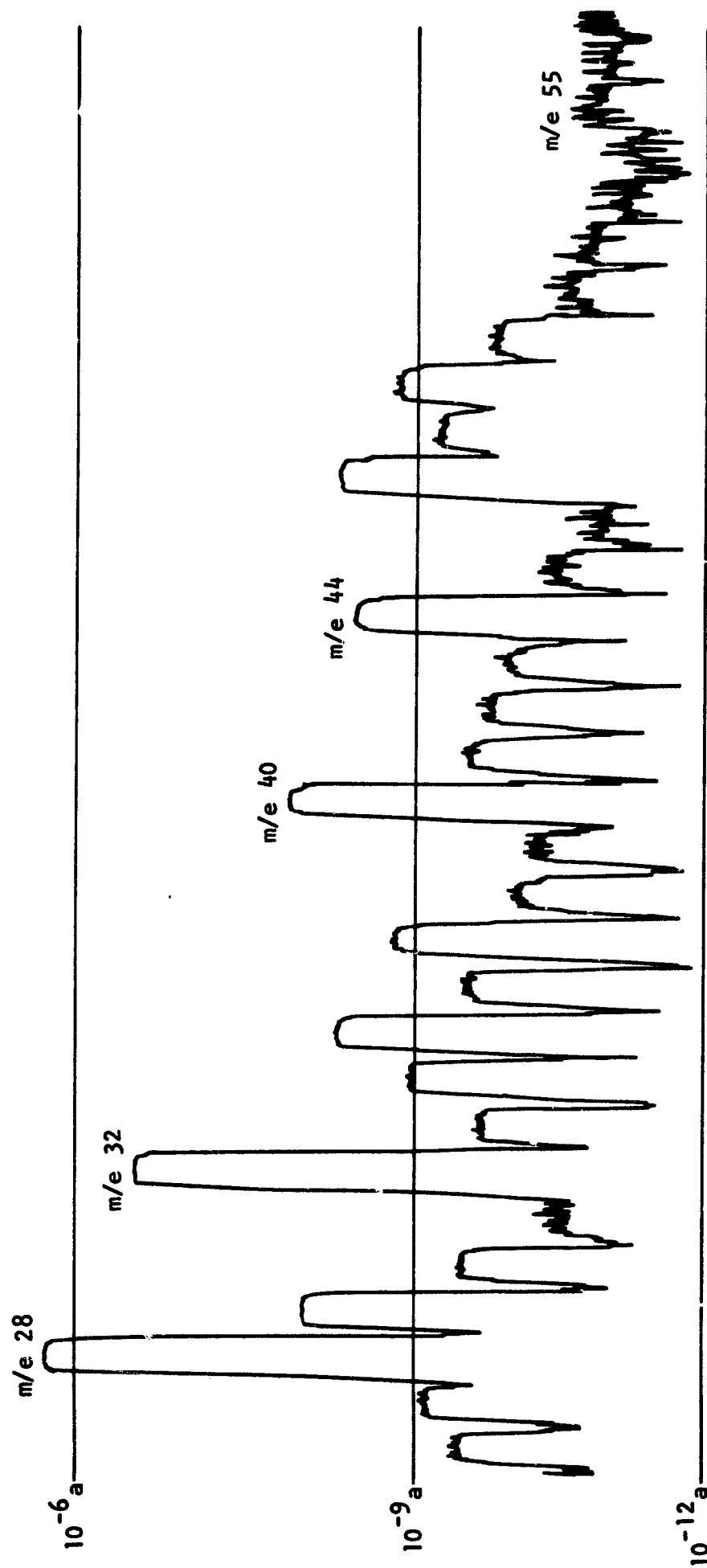


FIGURE 16. Mass Spectrum - m/e 26 to m/e 56

f 1.4 MHz

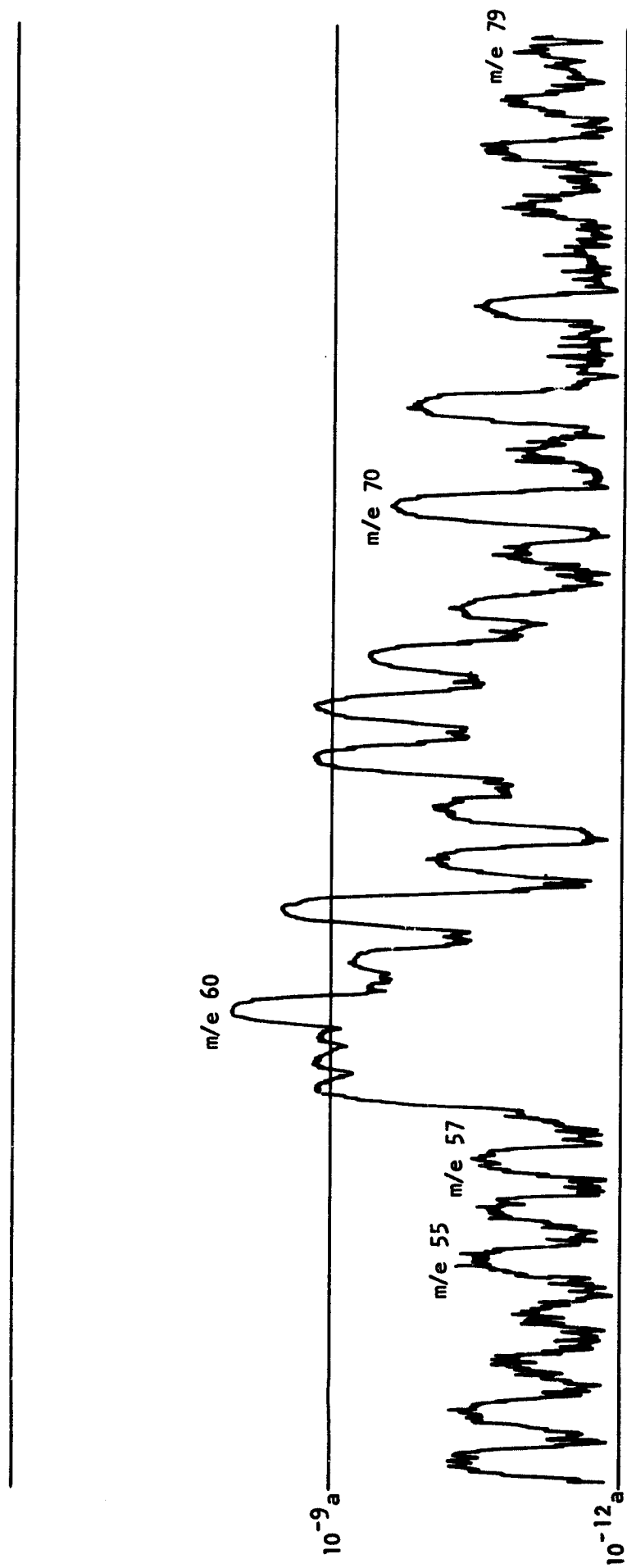


FIGURE 17. Mass Spectrum - m/e 51 to m/e 79

f 1.4 MHz

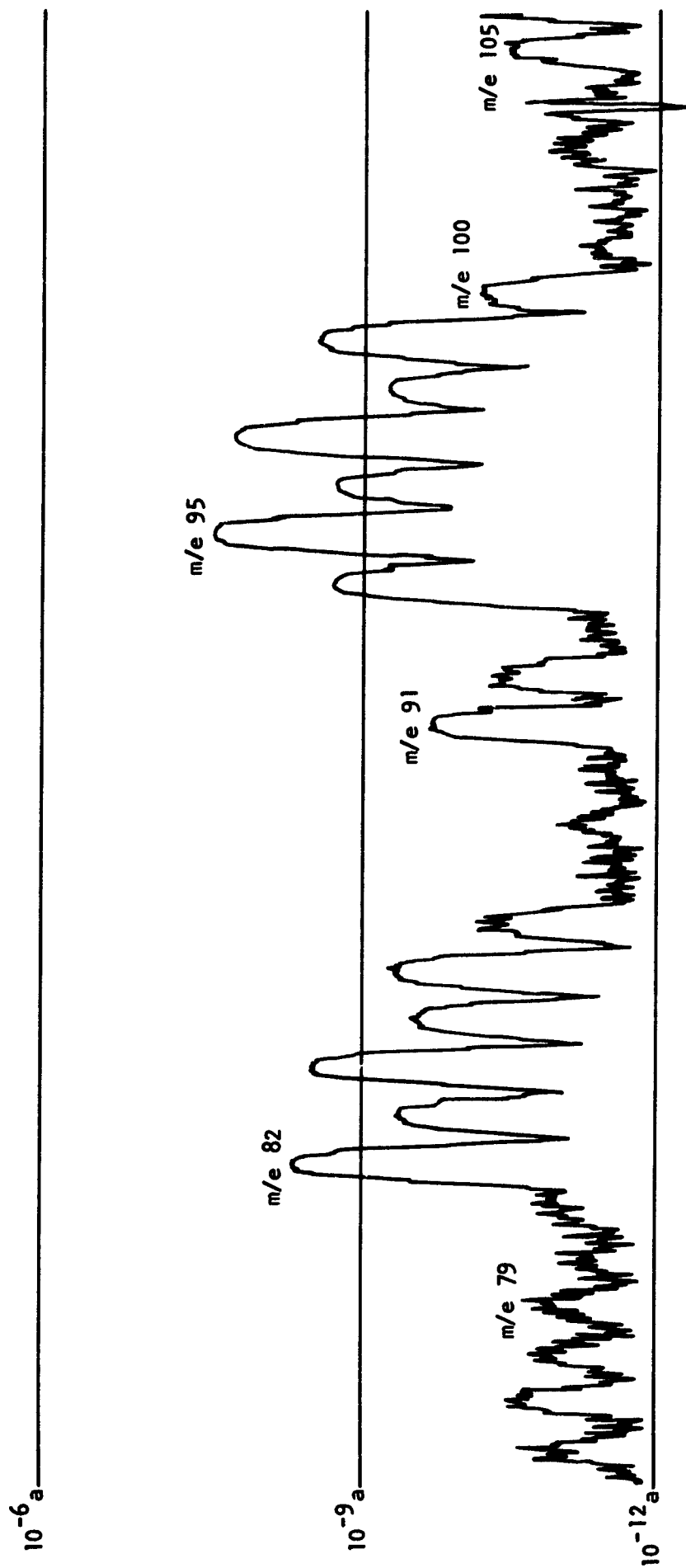


FIGURE 18. Mass Spectrum - m/e 76 to m/e 105

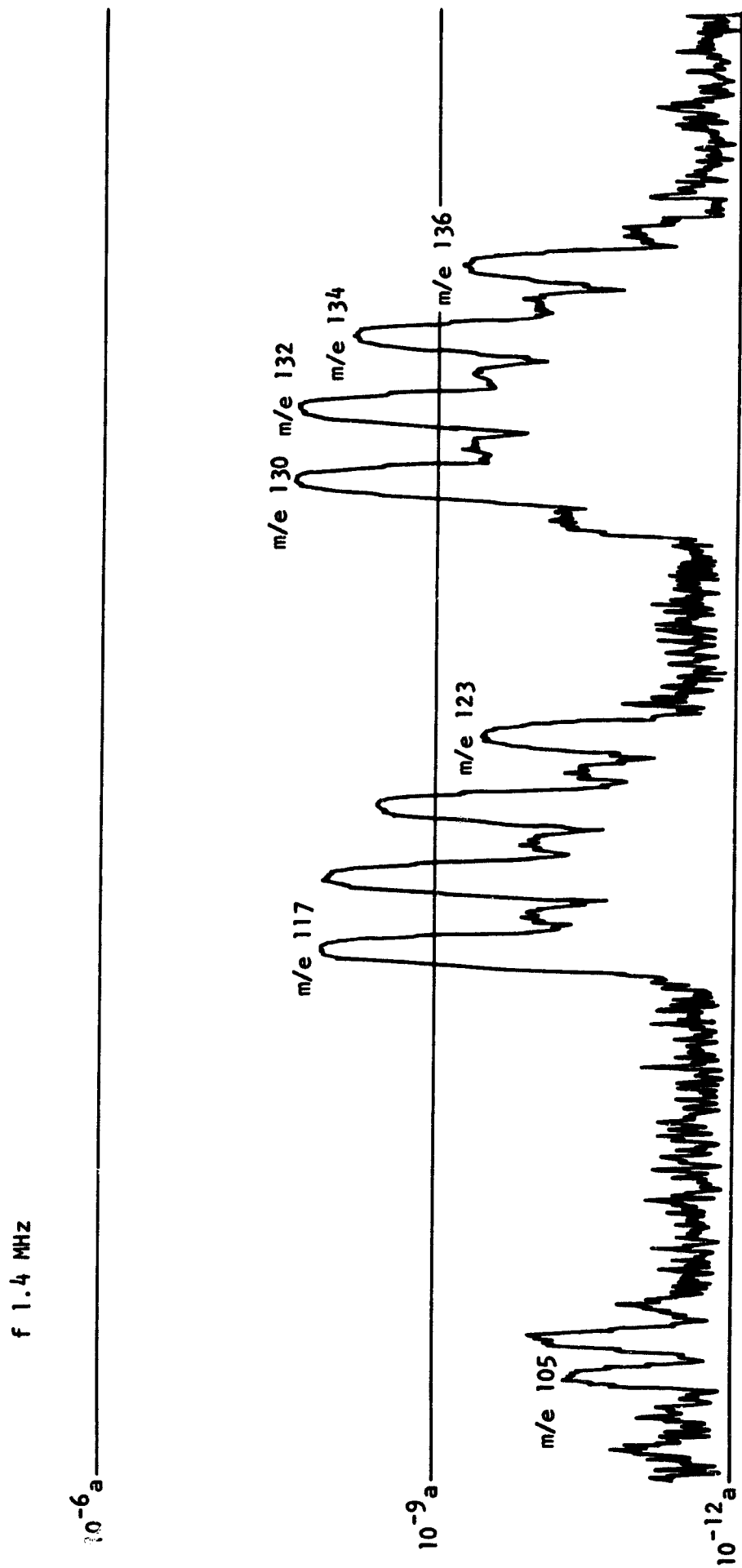


FIGURE 19. Mass Spectrum - m/e 103 to m/e 141

RESOLUTION
CARBON DIOXIDE - PROPANE MIX
 $M/\Delta M = 605$

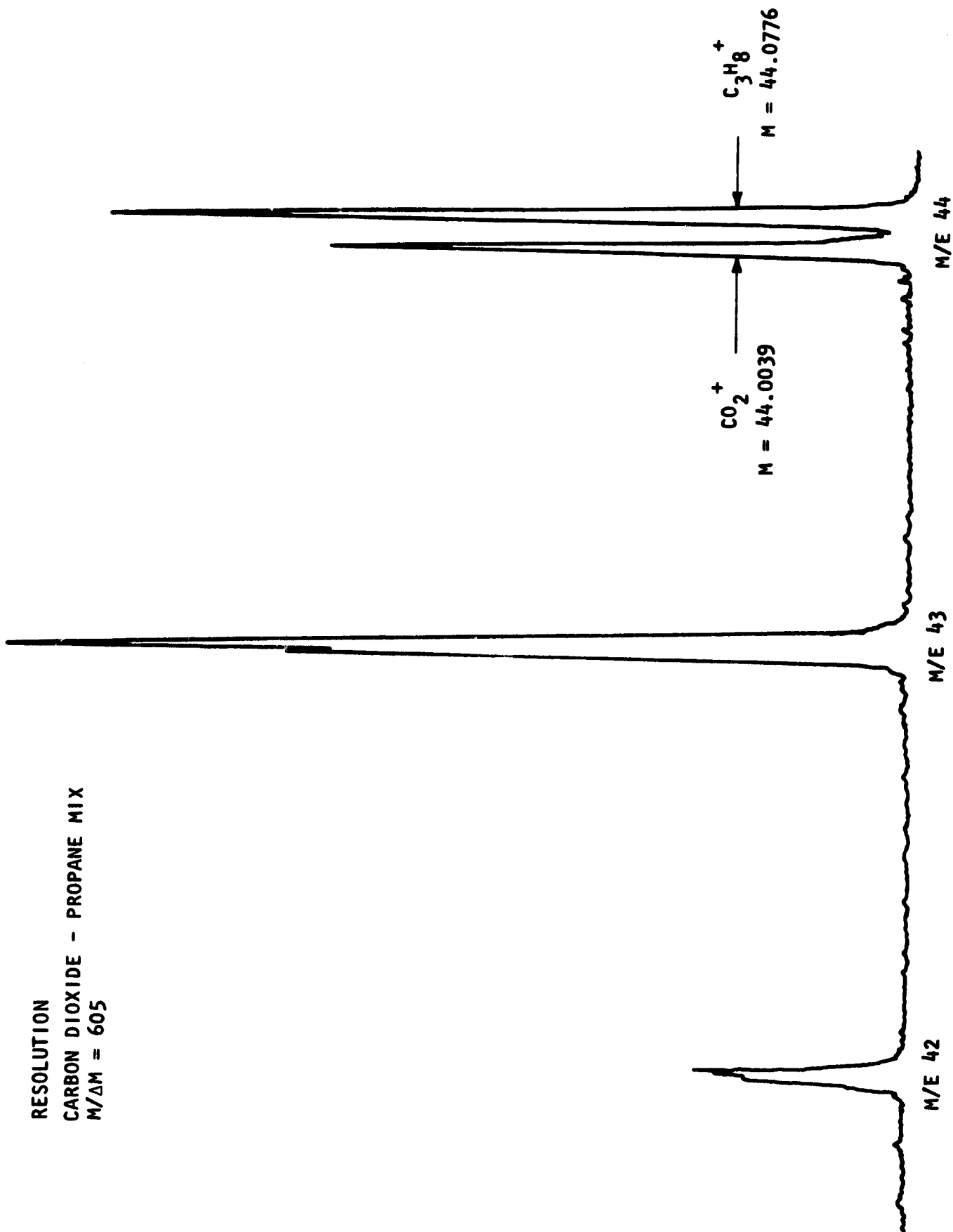


FIGURE 20. Separation of CO_2^+ and C_3H_8^+ Peaks

I_{an} 30 μ A
P_{SYST} 4.2 x 10⁻⁷ T
N₂ SAMPLE

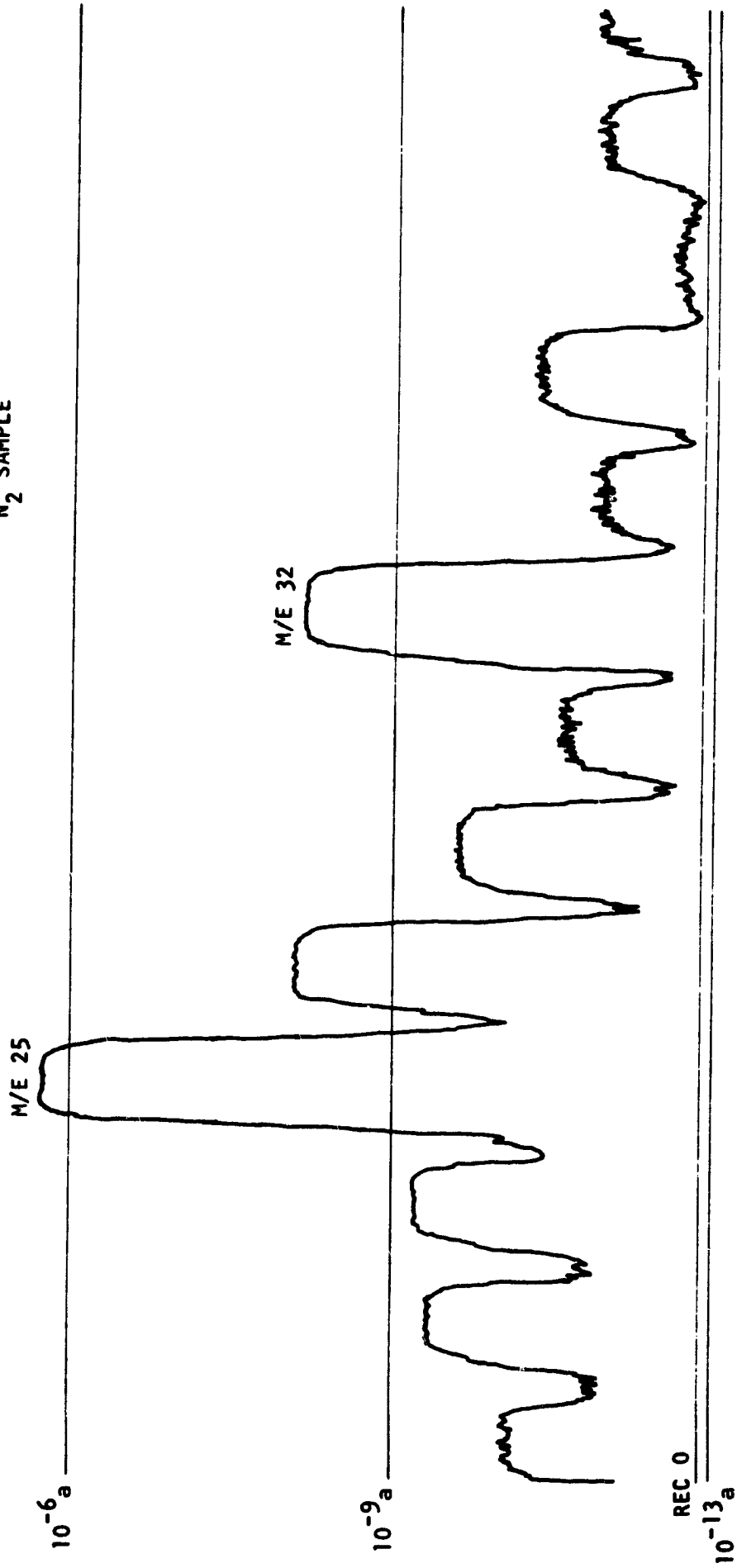


FIGURE 21. Dynamic Range After Weldup

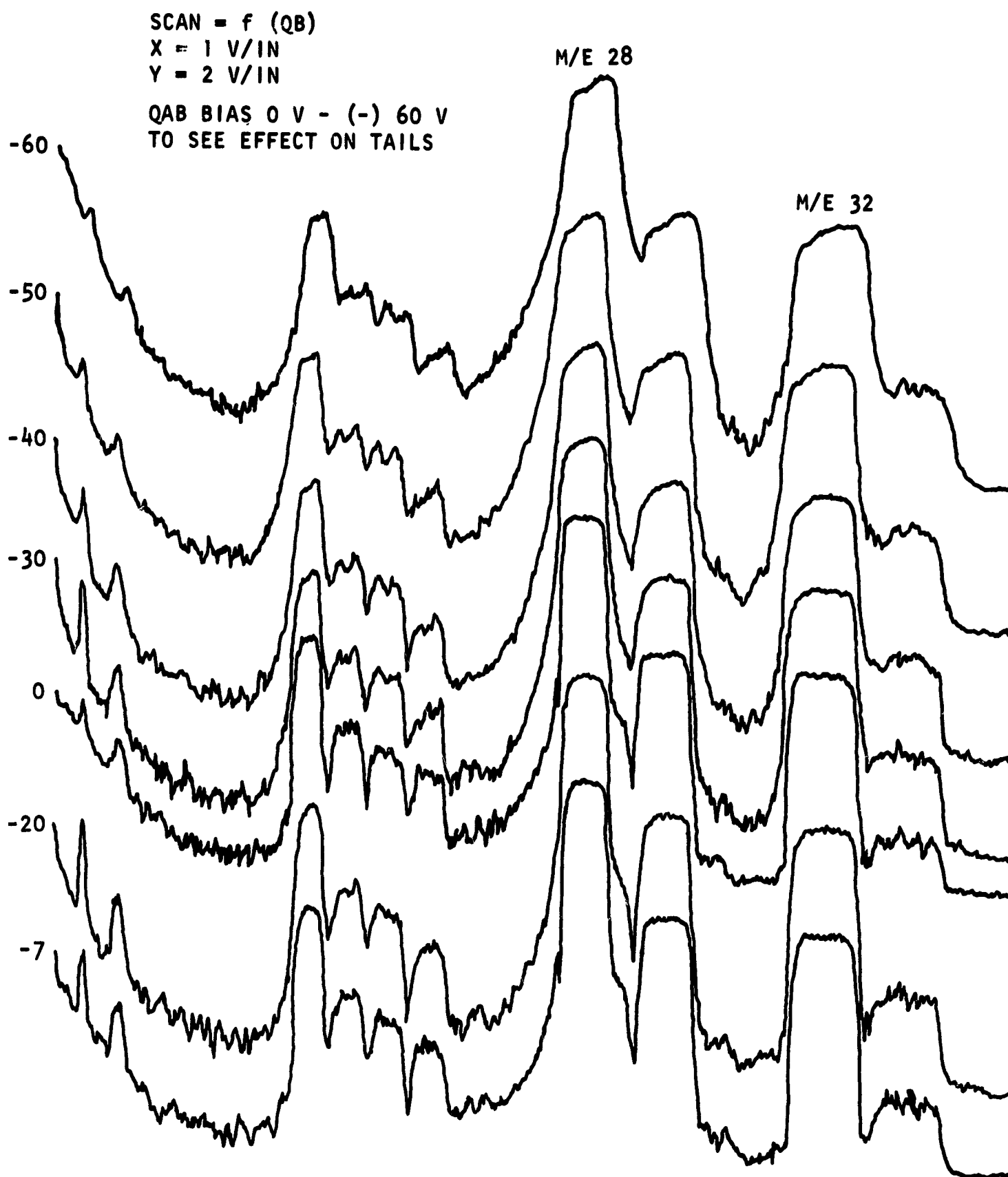
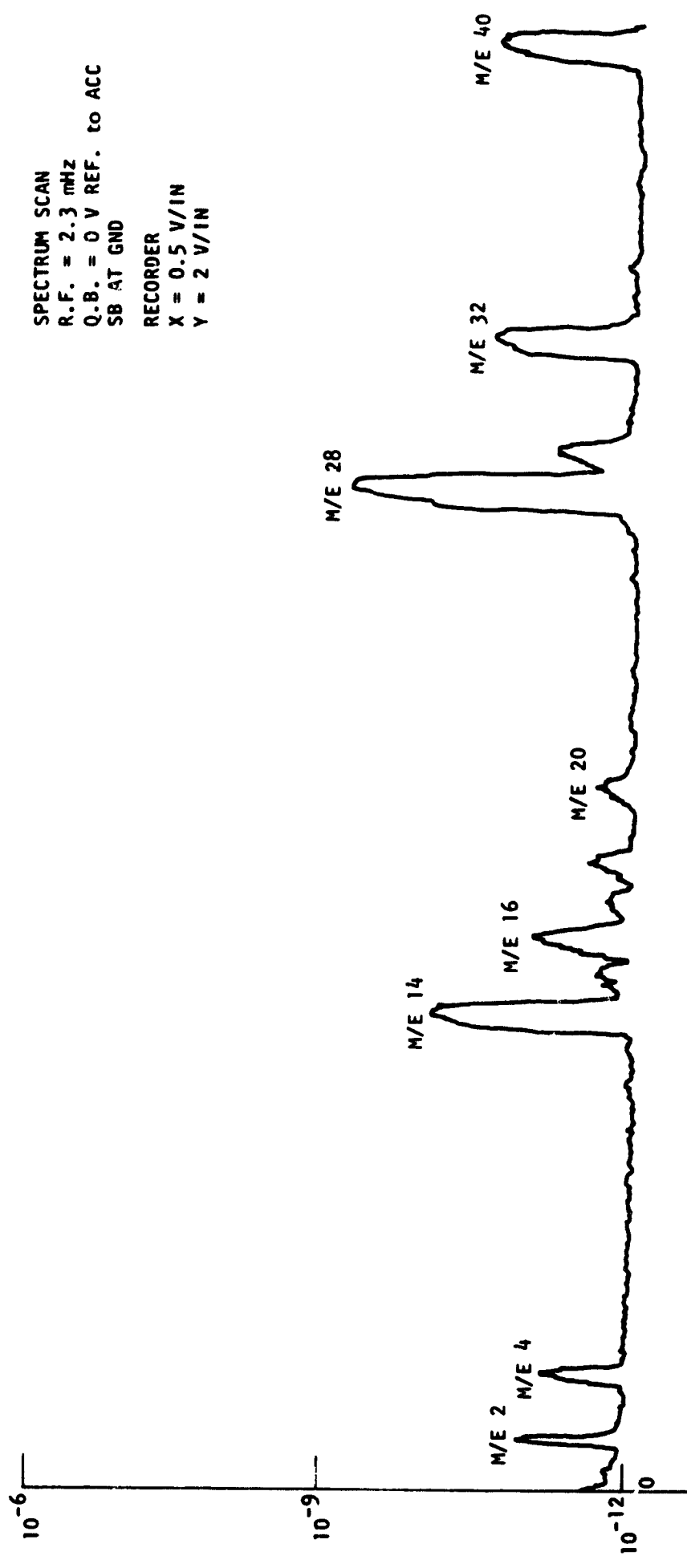


FIGURE 22. Quadrupole Bias Effect on Peak Tails



SPECTRUM SCAN
R.F. = 2.3 MHz
Q.B. = 0 V REF. to ACC
SB AT GND
RECORDER
X = 0.5 V/IN
Y = 2 V/IN

FIGURE 23. Open Ion Source Mass Spectrum

BACKGROUND ABOUT $\sim 85 \mu\text{A}$

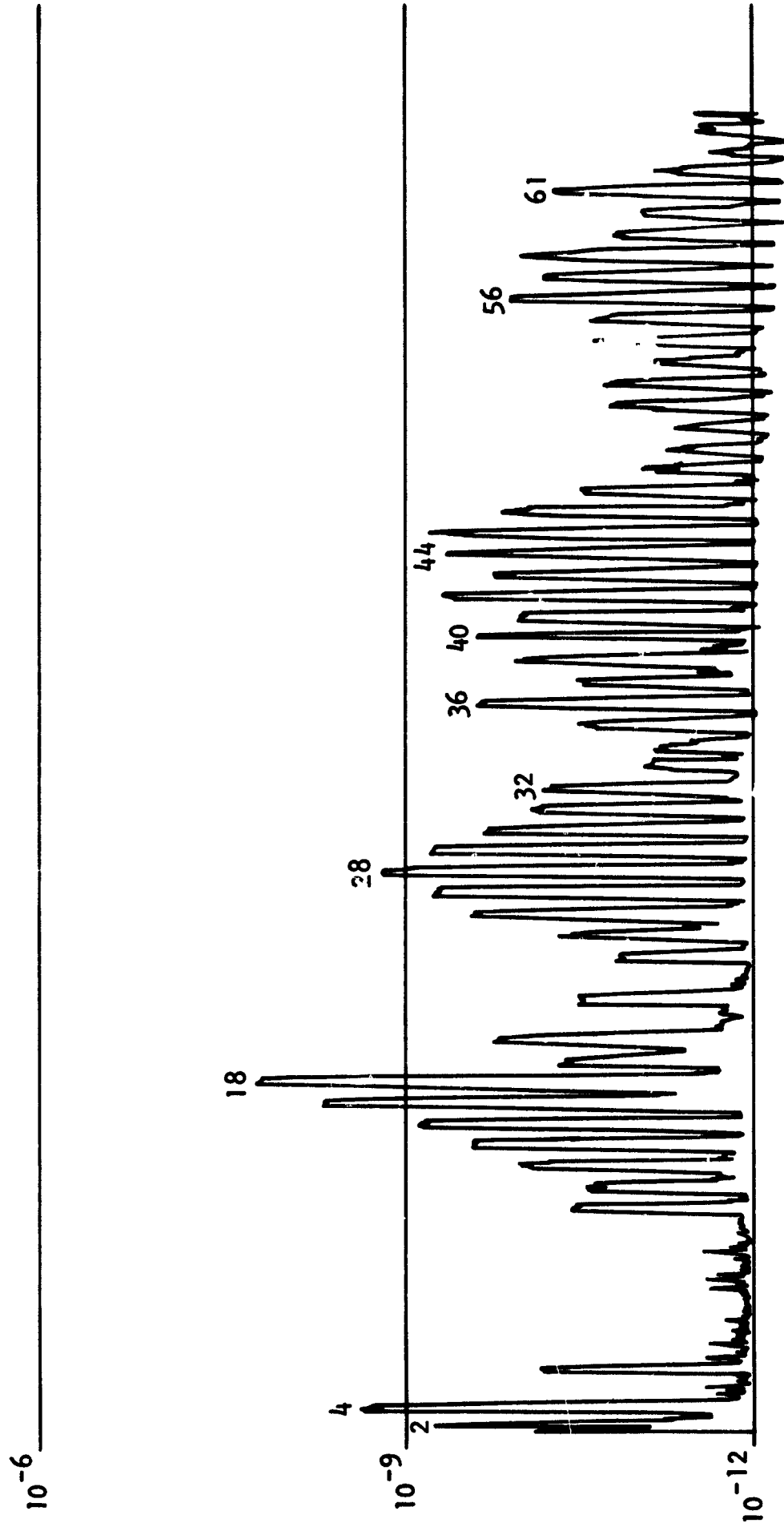


FIGURE 24. Background Spectrum Taken With S/N 001 at $\sim 2 \times 10^{-7}$ Torr

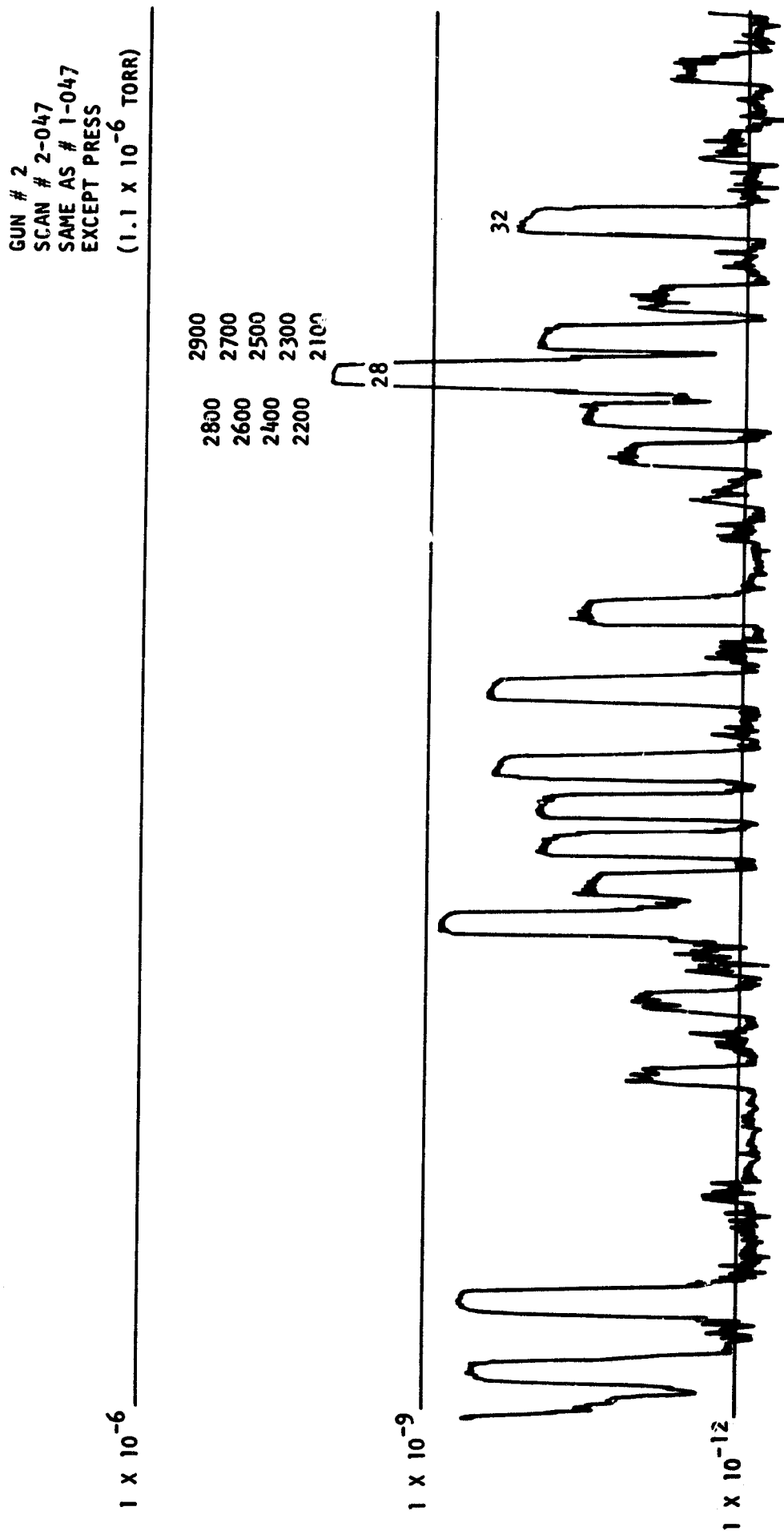


FIGURE 25. Typical Air Spectrum With -2000 Volts on the Electron Multiplier

GUN #2
 PR. = 1.1×10^{-6} TO R (N_2)
 SCAN #3-047
 H.V. = -2900 V
 (OTHER UNCHANGED)

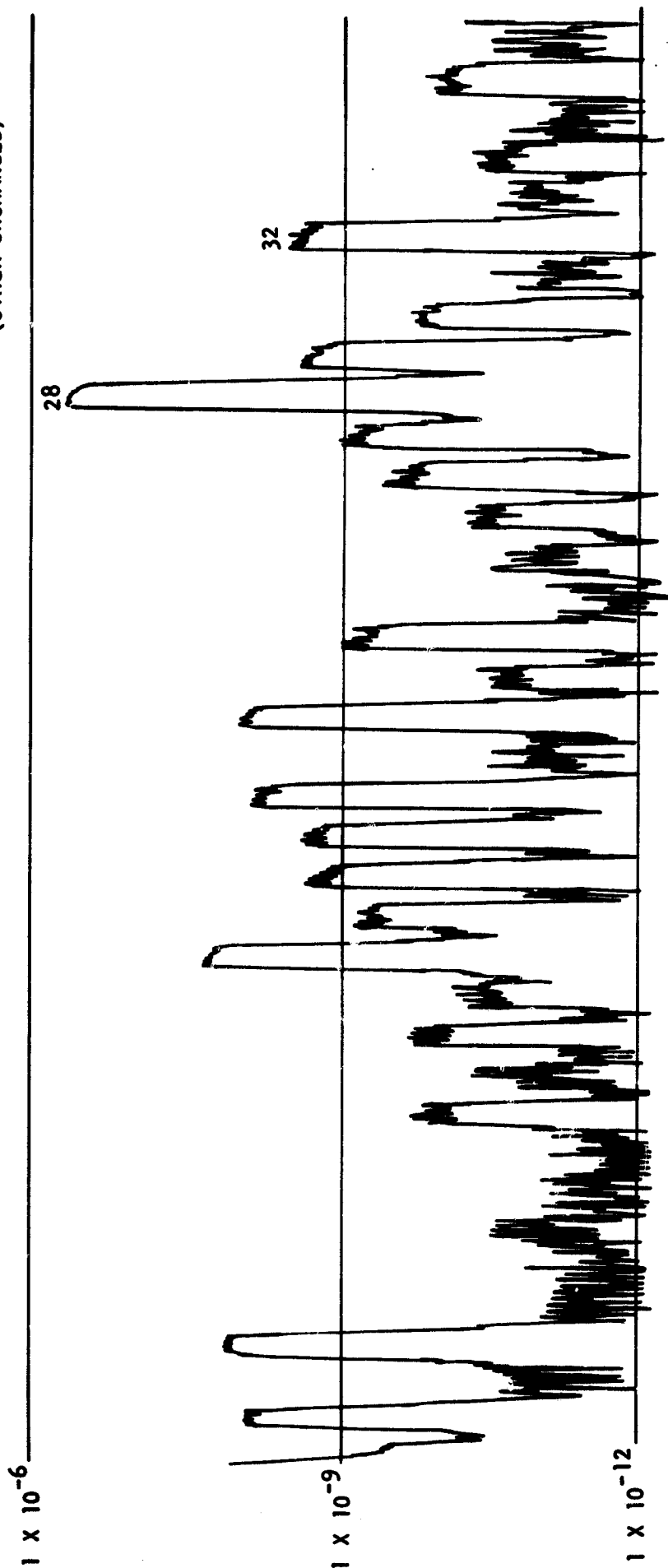


FIGURE 26. Typical Air Spectrum With -2900 Volts on the Electron Multiplier

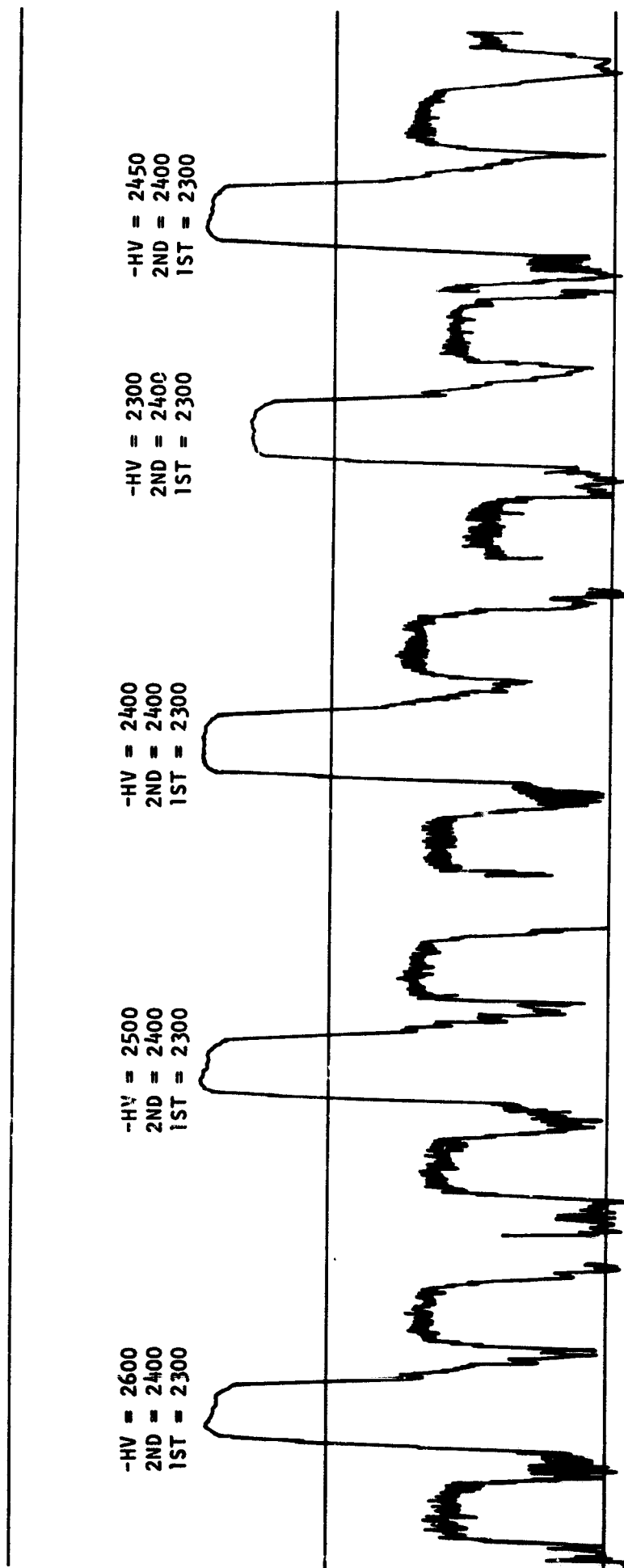


FIGURE 27. Scan No. 4-040, 2nd Window and -H.V. Voltage Affect Rods Normal

#3-040
2ND WINDOW & HV VOLTAGE AFFECT
RODS REVERSED

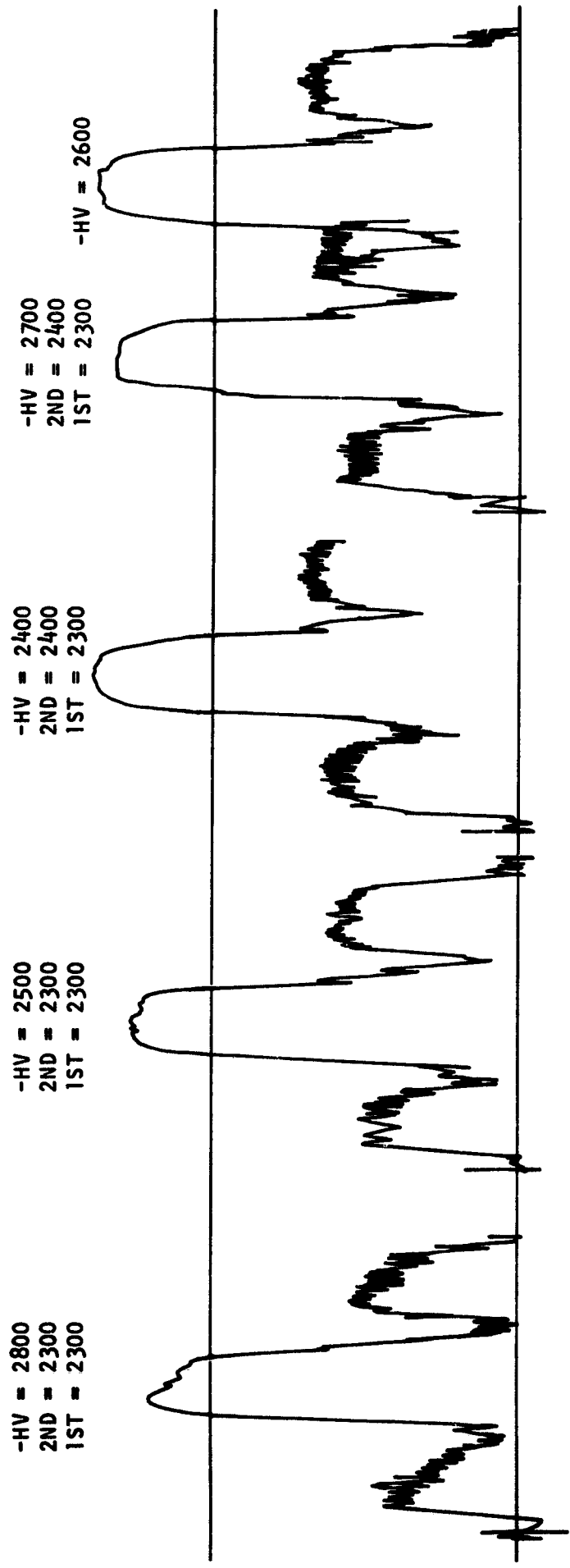


FIGURE 28. Scan No. 3-040, 2nd Window and H.V. Voltage Affect Rods Reversed

SCAN #1-041
1ST WINDOW AFFECT
QB @ ACC.
NOZZLE = 0 V

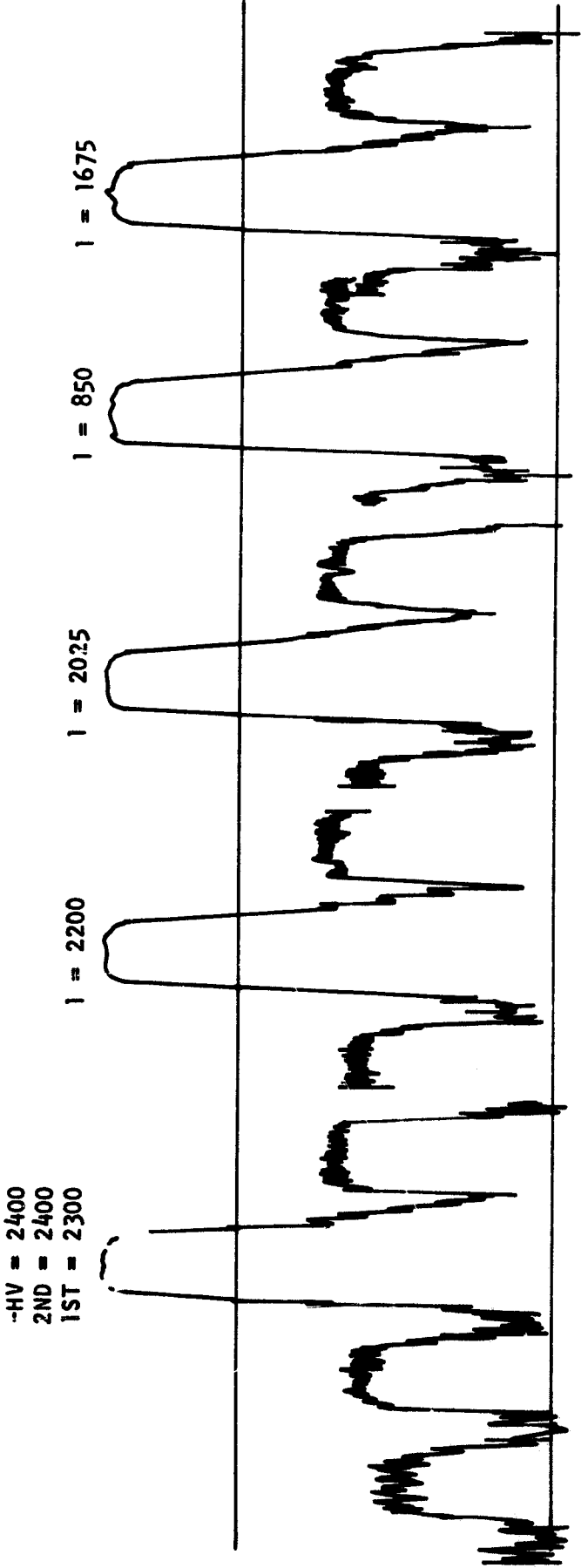


FIGURE 29. Scan No. 1-041, 1st Window Affect QB at ACC. Nozzle = 0 V

SCAN # 2-041
DEFLECTOR AFFECT

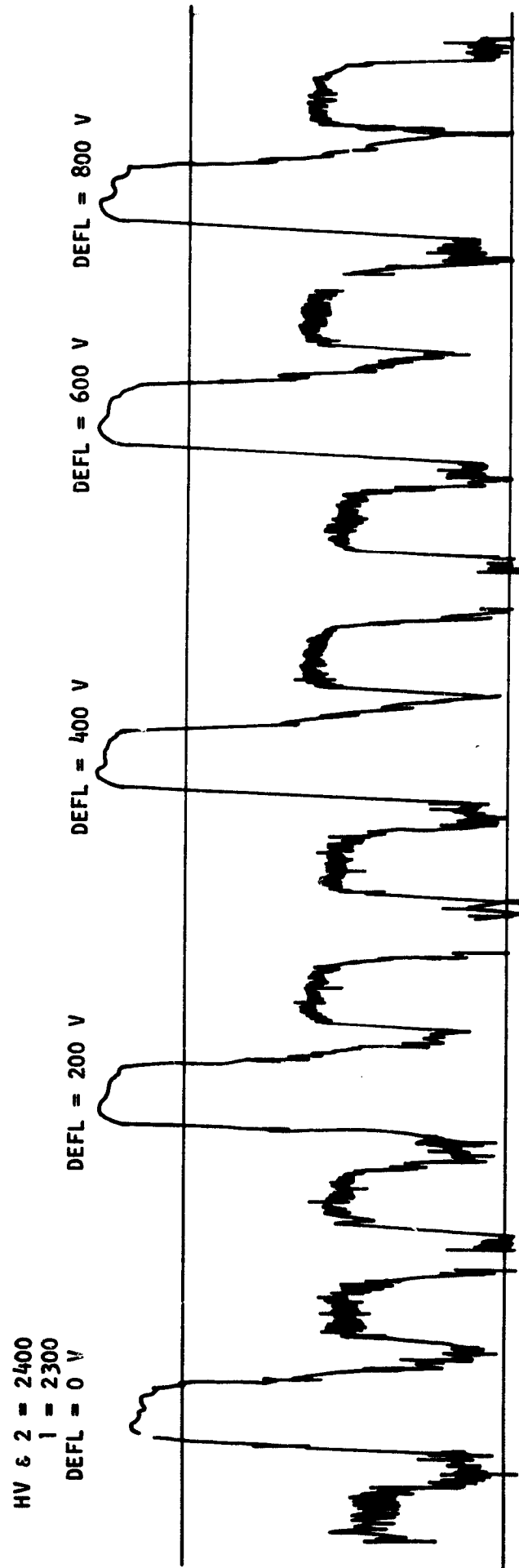


FIGURE 30. Scan No. 2-041, Deflector Affect

SCAN 3-041
HV & 2ND WIND AFFECT

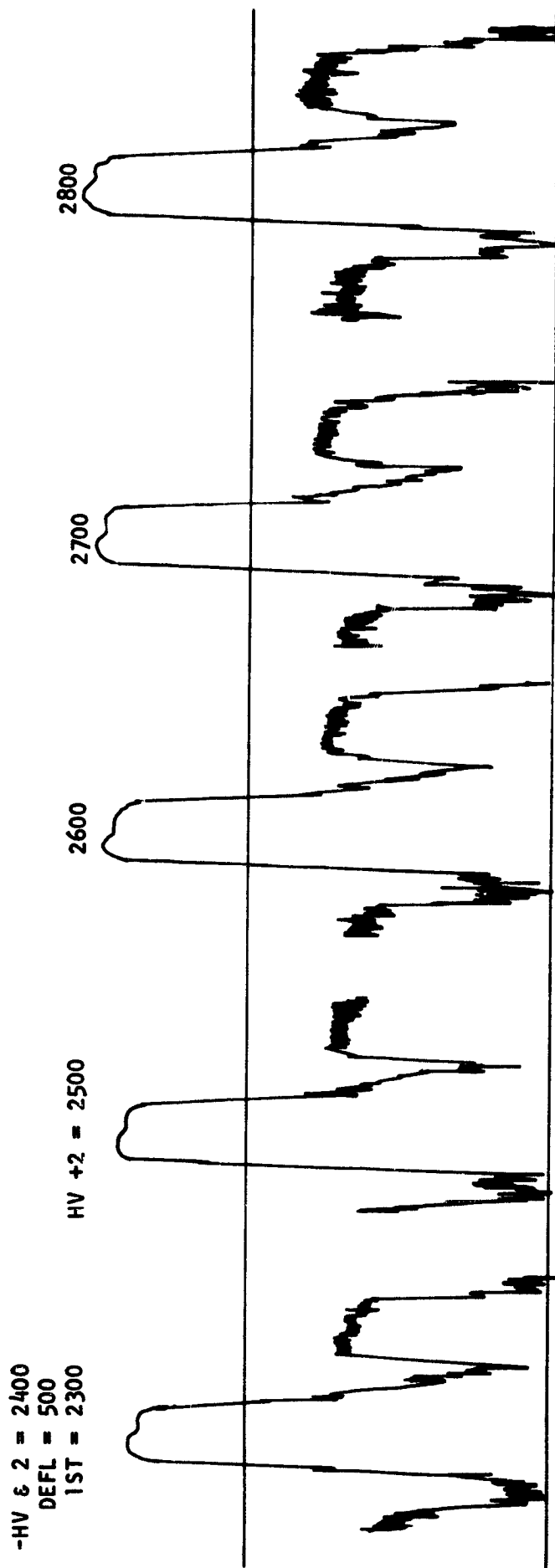


FIGURE 31. Scan 3-041, H.V. and 2nd Wind Affect

SCAN # 4-041
QB TEST

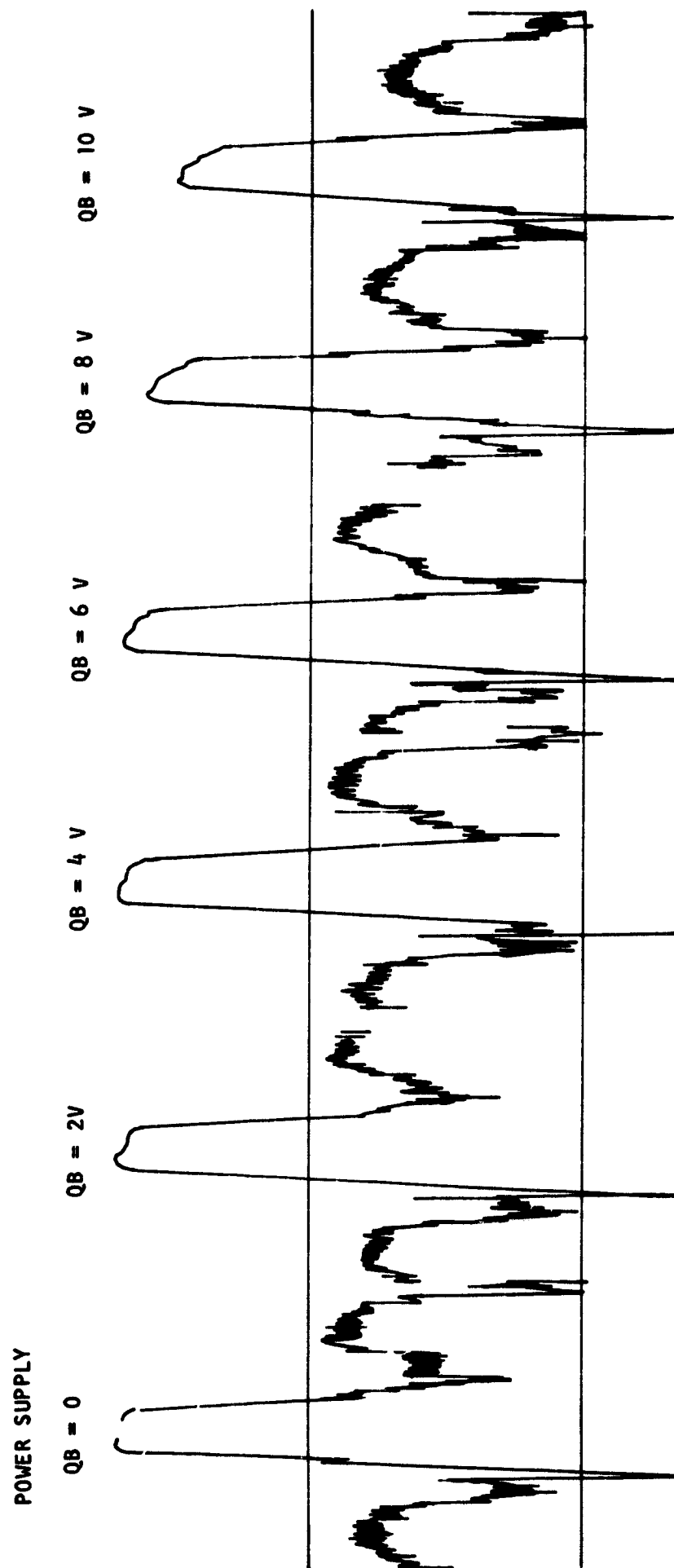


FIGURE 32. Scan No. 4-041, QB Test

LAST RUN BEFORE DELIVERY

GUN # 2	HV $\sim$ 2000
P $\sim$ 5 X 10 ⁻⁷	2ND $\sim$ 2000
ANODE 15 μ A	1ST $\sim$ 1850

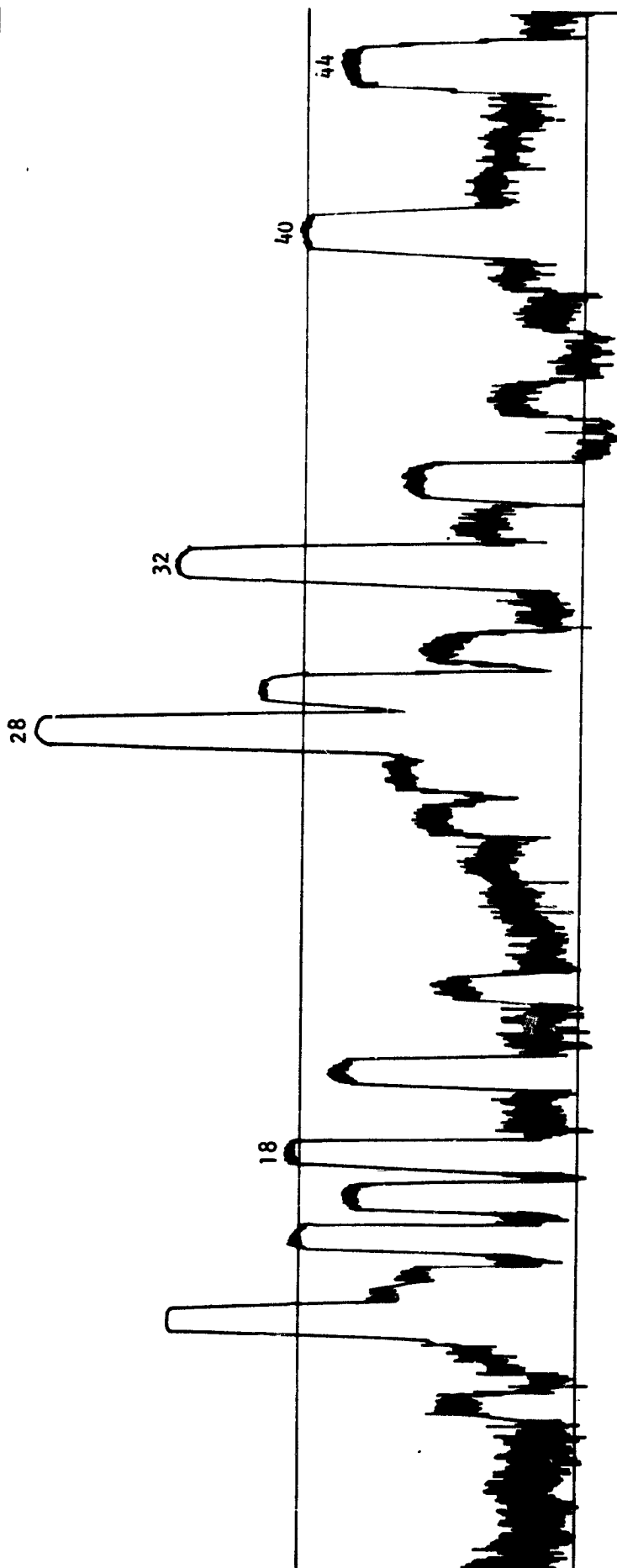


FIGURE 33. Last Run Before Delivery