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A STUDY OF THE INTRODUCTION OF IONS INTO THE REGION OF STRONG FIELDS WITHIN A QUADRUPOLE MASS SPECTROMETER

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ABSTRACT

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This project is a combined theoretical and experimental study of the introduction of ions into the strong fields of a quadrupole mass filter. Theoretical studies were made during the first quarter with the aid of a digital computer. Apparatus was assembled and preliminary experimental data were taken during the second quarter to verify the predictions made by the digital computer. The apparatus, procedures, and experimental data are described. Through the use of an additional set of electrodes near the entrance to the quadrupole it has been possible to increase the transport of ions through the instrument by factors of 10 to 100, without sacrifice in resolving power. This is accomplished by reducing the ratio of the dc to the ac potentials applied to the additional electrodes, as compared to the potentials applied to the quadrupole.

TABLE OF CONTENTS

LIST OF FIGURES

Figure

OBJECTIVES

The overall objectives of this contract are found in the Statement of Work of the contract and are simply stated as follows:

1. Conduct theoretical studies and laboratory experiments designed to improve the entrance conditions and appreciably increase the transmission efficiency of the filter.

2. Conduct a comprehensive analysis of the trajectories as the ions enter the filter such as can be made with the help of a digital computer.

3. Construct experimental models to take advantage of information gained from items 1 and 2.

4. Perform general experiments designed to demonstrate gains obtainable from the above.

5. Conduct experiments in which ions are introduced into the filter in bunches at specified phase angles.

6. Using a digital computer, compare the trajectories of ions in circular and hyperbolic quadrupole structures.

7. Construct apparatus to demonstrate the advantage found during the study of item 6, above.

INTRODUCTION

This report covers the work done by the Bell & Howell Research Center on NASA Contract NASW-1298, from 17 November 1965 through 17 February 1966 (the second quarter of the Contract).

This project is concerned with the introduction of ions into the region of strong fields in the quadrupole mass filter. During the first quarter of this contract a computer was used to study the problem and the proposed solution to the problem. The computer data substantiated that there is indeed a problem, and that the proposed solution is effective in eliminating it.

During the second quarter experimental apparatus was assembled and put into operation for the purpose of exploring the effectiveness of a proposed solution on an operating apparatus. Preliminary data have been obtained which show dramatically some of the improvements which result from an alteration of the electric fields in the entrance region.

THE PROBLEM

In the conventional quadrupole the fields are produced by potentials applied to one set of four electrodes. In this case the ratio of the dc to the ac fields is the same in the fringing region as it is within the quadrupole. Thus, for an ion whose working point is near the apex of the stability diagram for the conditions of full field within the quadrupole, the working point corresponding to the weaker fields in the fringing region lies in the y-unstable portion of the stability diagram. As the ion traverses the fringing field its working point moves along the scan line, as shown in Fig. 1.

All ions except those which enter the quadrupole very near the x-z plane, where the y-directed fields are zero, experience strong radial accelerations as they traverse the fringing field region. As a result, the radial impulse which an ion receives in this region causes it to have a high probability of striking one of the rods and being lost.

The problem, simply stated, is to improve the efficiency of transport of ions through the filter.

THE SOLUTION

The solution to the problem concerns the avoidance of the motion of the working point through the y-unstable portion of the stability diagram. This can be achieved by altering the ratio of the dc to the ac fields in the region of intermediate field strength. In order to avoid the undesired portion of the stability diagram, it is necessary that the ratio of the dc to the ac field in the fringe region be considerably smaller than the nominal 0.168 of the conventional quadrupole. This desired condition can be accomplished by the addition of one or more four-electrode sets at the entrance end of the quadrupole, and by energizing them with potentials such that the ratio of the dc to the ac is considerably less than the 0.168 value of the mass analyzing portion of the quadrupole. In fact, the electrodes which form the field at the entrance to the quadrupole may be energized with ac potentials only. In order to minimize the transient which the ions experience as they enter the ac field, these ac potentials may be of low value.

The application of the very low or zero dc potentials to the entrance electrodes has been termed "delayed dc ramp." Although discrete potentials are applied to the electrodes, the fields near the axis of the instrument rise gradually, in ramp fashion, as the ions enter the quadrupole.

By delaying the application of dc potentials, relative to the ac potentials, (delayed along the z- or instrument-axis) the working point is caused to move through the stable portion of the diagram and the undesired radially directed impulse is avoided. The paths taken by the working point in the conventional and in the modified quadrupole are shown in Fig. 1.

APPARATUS

The apparatus used for the experiments described in this report consists of an assembly of different components each of which was designed, developed, and fabricated at the Bell & Howell Research Center with Corporate funds prior to the initiation of this contract. The assembled apparatus is shown in block diagram form in Fig. 3.

The Quadrupole

The diameter of the rods in the quadrupole is that used for previously designed units for space exploration, 0.600 inches. However, the rods, made of ceramic, are unusually long (18 inches), because the quadrupole was designed for other experiments, which were sponsored by Bell & Howell. However, its versatility makes it very suitable for the experiments of this project.

Conductive surfaces have been formed on the ceramic rods by gold plating. Sufficient surface areas have been removed to form four 0.600-inch long segments at the ion entrance end of each rod. These segments, unfortunately, are separated by exposed rings of bare ceramic on which electrical charges may accumulate.

Electrical connections inside the quadrupole connect corresponding segments of opposing rod pairs, and leads from each pair are brought through the vacuum wall. Thus, the chosen values of dc and ac potentials may be applied to each pair of leads.

Ion Source and Entrance Geometry

The ion source is of circular geometry, with the density of ionizing electrons being a maximum in the vicinity of the axis. Potentials applied to coaxial, annular rings produce fields which accelerate the ions in the axial direction, toward the quadrupole.

The geometry of the source and the entrance aperture is shown in Fig. 4. For the purposes of these tests the source is spaced from the entrance aperture to provide a more uniform density of incident ions across the aperture and to better collimate the ion beam.

Radio-Frequency Oscillator

The self-excited push-pull oscillator provides 1.16 MHz when loaded with the total capacity of the rods and segments. Dc rod potentials are obtained by rectification of the ac potentials. The ac and the dc voltages applied to the short rod segments are independently adjustable.

Mass scanning is accomplished by varying all potentials in proportion. The incremental potential variations required for the desired mass scan are controlled by the saw-tooth sweep voltage (horizontal output) of the cathode ray oscillograph. Thus, the displayed spectrum is synchronized. The mass spectrum was scanned, repetitively, at a rate of ten scans per second.

Mass resolution is adjusted by varying the ratio of the dc to the ac potentials.

Electrical Connections to Quadrupole

In Fig. 5 is shown the manner in which the electrical connections to the short rod segment pairs are changed. Small pin sockets are attached to the external ends of the feed-through connectors and corresponding sets are attached to convenient points along the quadrupole rod supply busses. Thus, the segmented rod quadrupole can be made to perform in the conventional manner by inserting jumper wires into the appropriate sockets, or it can be made to perform in the improved manner by substituting series capacitors for the jumper wires at the entrance segment pairs. The capacitors used in these experiments are air dielectric. Each segment pair is permanently connected to ground through a 32-megohm resistor in order to place the dc potential of the segment pair at zero when a series capacitor is used.

Secondary Emission Multiplier

A "venetian-blind" type of secondary emission multiplier (C-70120E made by RCA) using beryllium-copper dynodes is used to amplify the current from the quadrupole. It has a measured gain of between 10^5 and 10^6 when supplied with -4000 volts.

EXPERIMENTAL PROCEDURE

A comparison was made during each of the tests between the performance of the segmented rod quadrupole when connected conventionally and the performance of the same unit connected in such a way that the ac and dc voltages on the entrance segments could be varied with respect to those of the long rod segments. All other operating conditions remained constant during the period of the testing.

The spectrum of krypton was used for all measurements. It presents two nearly equal-sized peaks at masses 82 and 83. Further, its spectrum falls in a mass range in which there are no interfering background peaks.

Performance of the quadrupole in the two modes of operation was compared by observing the transmitted ion currents at comparable resolving powers for each mode. Two different methods were used in obtaining data at comparable resolving powers. In one, the dc/ac ratio was used as an independent parameter while the current in the mass 84 peak and the resolving power were observed. In the other, the valley between the equal-height peaks at masses 82 and 83 was made to be a constant fraction of these peak heights.

EXPERIMENTAL DATA

General Observations

Several methods of effecting a delay in the dc ramp were tried in making the comparisons between the conventional mode of operation and that of the delayed dc ramp. Varying amounts of dc potential were applied to the entrance segment pairs to approximate the conditions fed to the computer. As predicted by the computer studies, it was observed that it makes little difference just how the dc ramp was tailored so long as the working point remains below the y-stability limit. For this reason, zero dc volts was used for the tests.

Lowering the ac voltage on the first segment pair improves the ion transmission for all manners of operation. Potentials of about 20% of the full ac rod voltages are near optimum.

Varying the dc and ac voltages on the second, third, and fourth segment pairs was also tried but no improvement was observed over the results obtained with just the first segment pair.

Peak Height vs Resolving Power, as Measured at Half Peak Height

The data of prime interest is the relation between peak height and resolving power. The independent parameter which influences both peak height and resolving power is the ratio of the dc to the ac potentials applied to the quadrupole. However, the manner in which they depend upon this ratio is a function of other operating parameters. Hence, the independent operational variable, dc to ac potential ratio, is used as a parameter to obtain the dependence of transmitted ion currents upon resolving power for each of the two modes of operation. The resulting data are presented in Fig. 6.

The data of Fig. 6 present the intensity of the krypton mass 84 peak as a function of the resolving power for the conventional quadrupole mode of operation and for the delayed dc ramp mode. The resolving power is calculated from the width of the peak at the half-height point, and is given by the formula:

Resolving power = 84 (distance between adjacent peaks/peak width)

As is to be expected, the ratio of the transmitted ion currents obtained in the two modes of operation increases at higher resolving powers. Fig. 7 relates this ratio to the resolving power. At a resolving power of 1,000, the collector current is 50 times higher in the delayed dc ramp mode than it is in the conventional mode, with all other operating parameters remaining unchanged.

Peak Height at Comparable Resolving Powers, as Determined by Normalized Valley-to-Peak Ratios

The height of the valley between adjacent peaks is a measure of the current in the tails of the peaks. During our preliminary exploration of the advantages to be obtained through the use of the delayed dc ramp, this criterion was used in making the comparisons. That is, the dc/ac potential ratio was adjusted to give a valley between the mass 82 and 83 peaks which was a chosen fraction of the height of the peaks. This adjustment was made for each mode of operation, and the transmitted ion currents compared.

Because this criterion for obtaining comparable performance for each mode is not well related to resolving power, or to some other quality-of-performance factor, the data are presented in tabular form, in Table I.

TABLE I

	Ion Current at the Collector		
Valley-to-Peak Ratio in %	Conventional	Delayed dc Ramp	Ratio of Current in the Two Modes
10	5.7	98	14.6
5	3.3	90	27
2.5	0.6	72	119

It is interesting to note that the increase of the ratio of the currents for operating conditions which produce lower valleys is obtained primarily from the decrease in the current as the resolving power is increased in the conventional mode of operation. That the corresponding change in the current for the delayed dc ramp mode is much smaller indicates that the peak is much better defined in this mode; the intensity of the current in the "tails" is much lower.

Comparison of Peak Shapes Obtained in Conventional and Delayed dc Modes of Operation

The double set of peaks shown in Fig. 8 well illustrates some of the advantages obtainable with the altered fields. The current scale differs by a factor of 10 in the two sets. The higher currents are obtained in the delayed dc ramp mode of operation. The peak at the highest current represents a resolving power of about 90 at the 5% points in each case.

As the dc to ac ratio was increased, families of peaks were produced. In each case (conventional and delayed dc ramp) the peak height is attenuated by this increase. In the case of the conventional quadrupole there is little narrowing of the peak at the 5% level. That is, the peak height decreases with only small increases in resolving power. For the case of the delayed ramp, each increase in dc/ac ratio results in a narrowing of the peak, at all levels. This represents an increase in resolving power. This fact is of prime importance.

Reference to the sets of peaks in Fig. 8 shows that the attenuation of current as the resolving power is increased occurs at a much faster rate in the conventional quadrupole than in the delayed dc ramp quadrupole. Although the data are too sparse to support any given number for this ratio, they suffice to show that an additional factor of 10 may be obtained at high resolving powers. This factor of 10 is to be multiplied by the factor of 10 in the attenuation of the gain used for the delayed dc ramp mode when the data of Fig. 8 were obtained.

The modulation of the tops of the peaks is anomalous. Perhaps it is due to the build-up of charges on the insulated portions of the ceramic rods, between the segments.

CONCLUSIONS

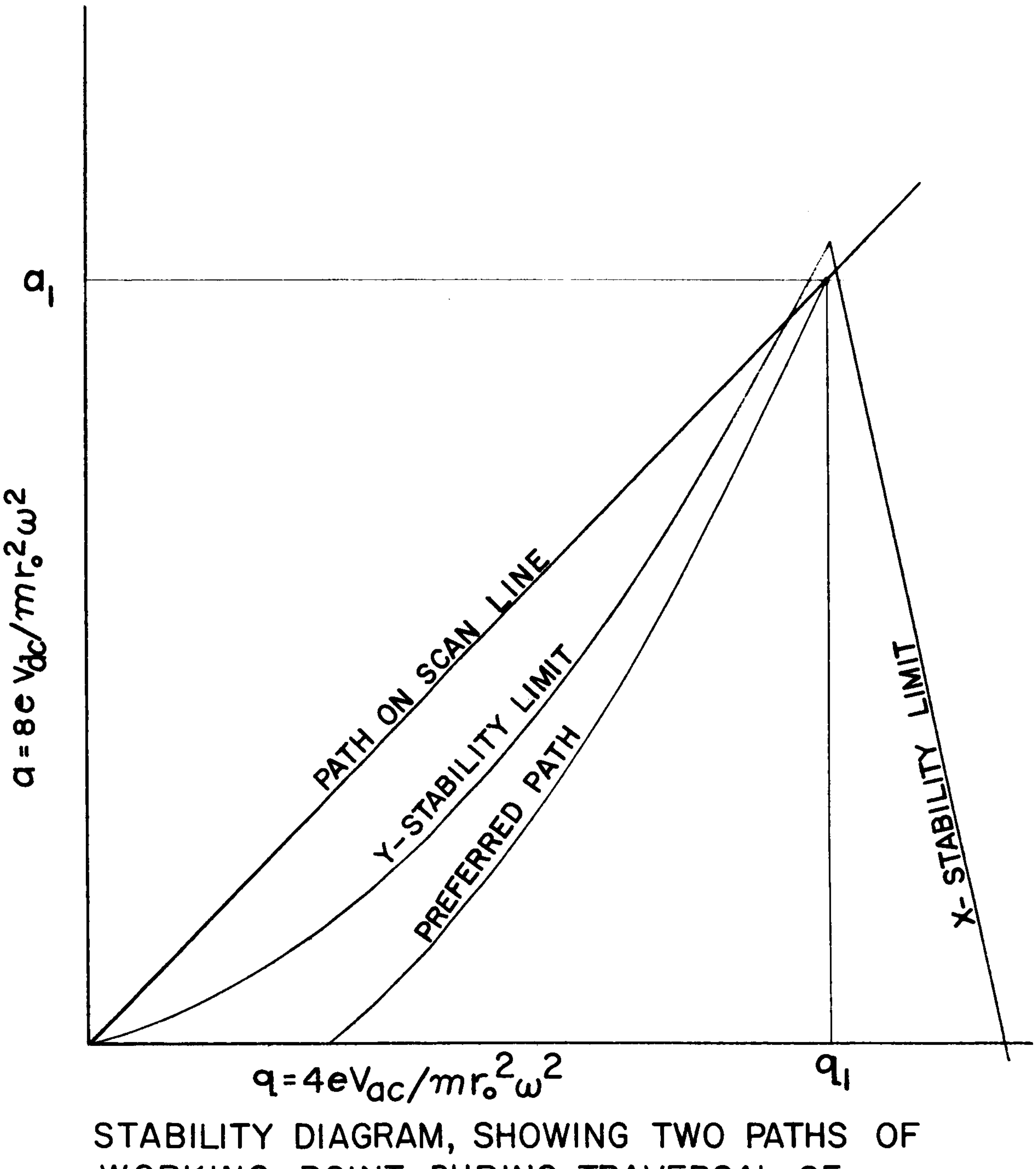
The predictions of the first quarter's computer study have been verified, in principle, in the laboratory. Through the use of an additional set of electrodes (short rod segments) at the entrance end of the quadrupole, it has been possible to demonstrate some of the potentialities of the proposed solution to the problem of introducing ions into the quadrupole. This has been accomplished by greatly reducing the ratio of the dc to ac potentials applied to the short rod segments, relative to those of the quadrupole.

The improvement in performance is most apparent in the increased ion current at the collector under conditions of high resolving power. This results largely through the avoidance of the huge loss of ions due to their traversal of the fringing electric fields. The current at the collector is increased by factors of 10 to 100 without loss of resolving power through the application of a delayed dc ramp at the entrance end of the quadrupole.

PROPOSED WORK DURING THE THIRD QUARTER

During the third quarter computer studies will be resumed. This time the computer will be used to evaluate the difference in the operation of a quadrupole structure with hyperbolic field-forming surfaces and a quadrupole with the more usual round surfaces.

Refinements will be made in the apparatus used to study the effectiveness of the delayed dc ramp.



STABILITY DIAGRAM, SHOWING TWO PATHS OF WORKING POINT DURING TRAVERSAL OF FRINGING FIELD.

FIGURE 1

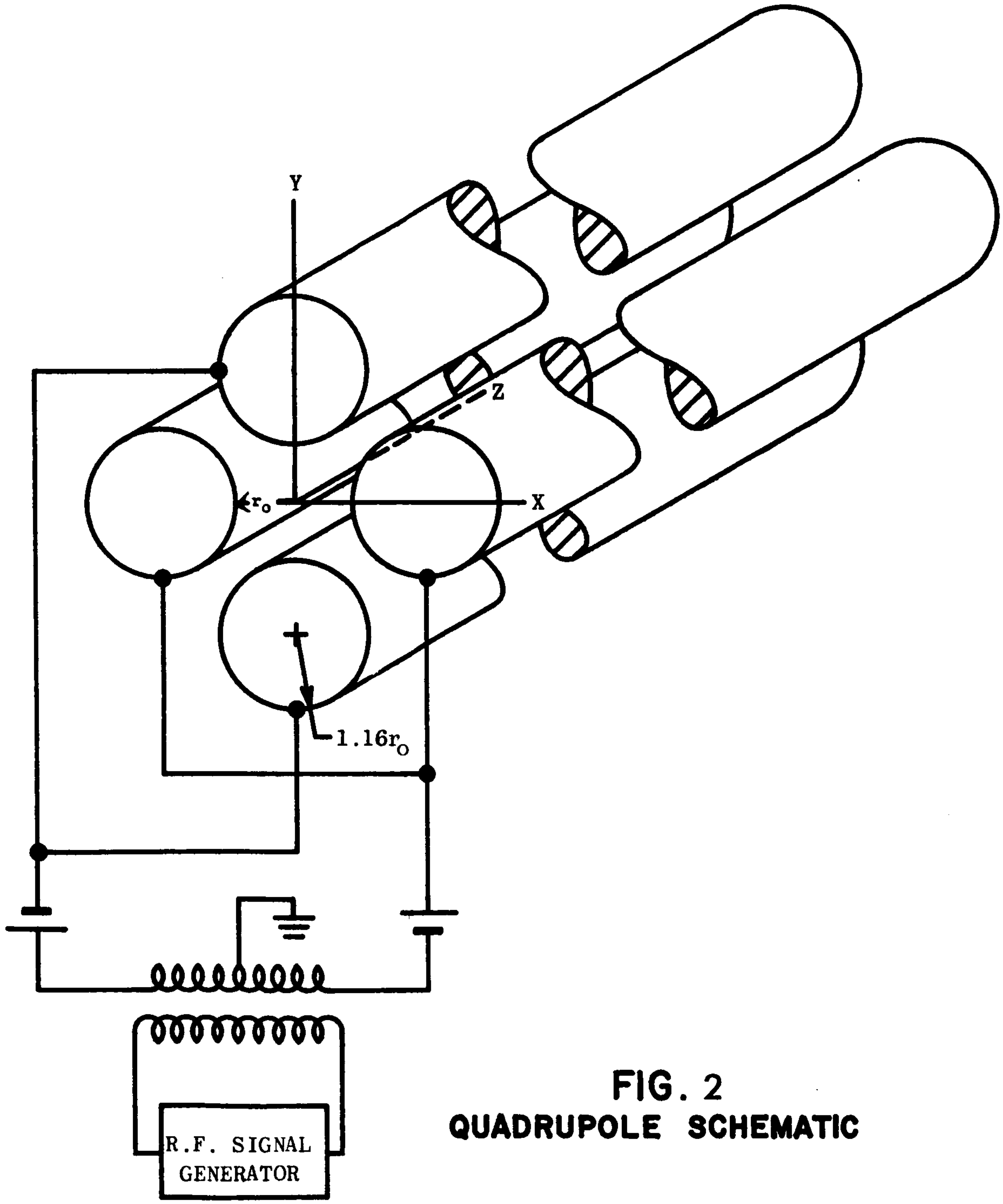


FIG. 2
QUADRUPOLE SCHEMATIC

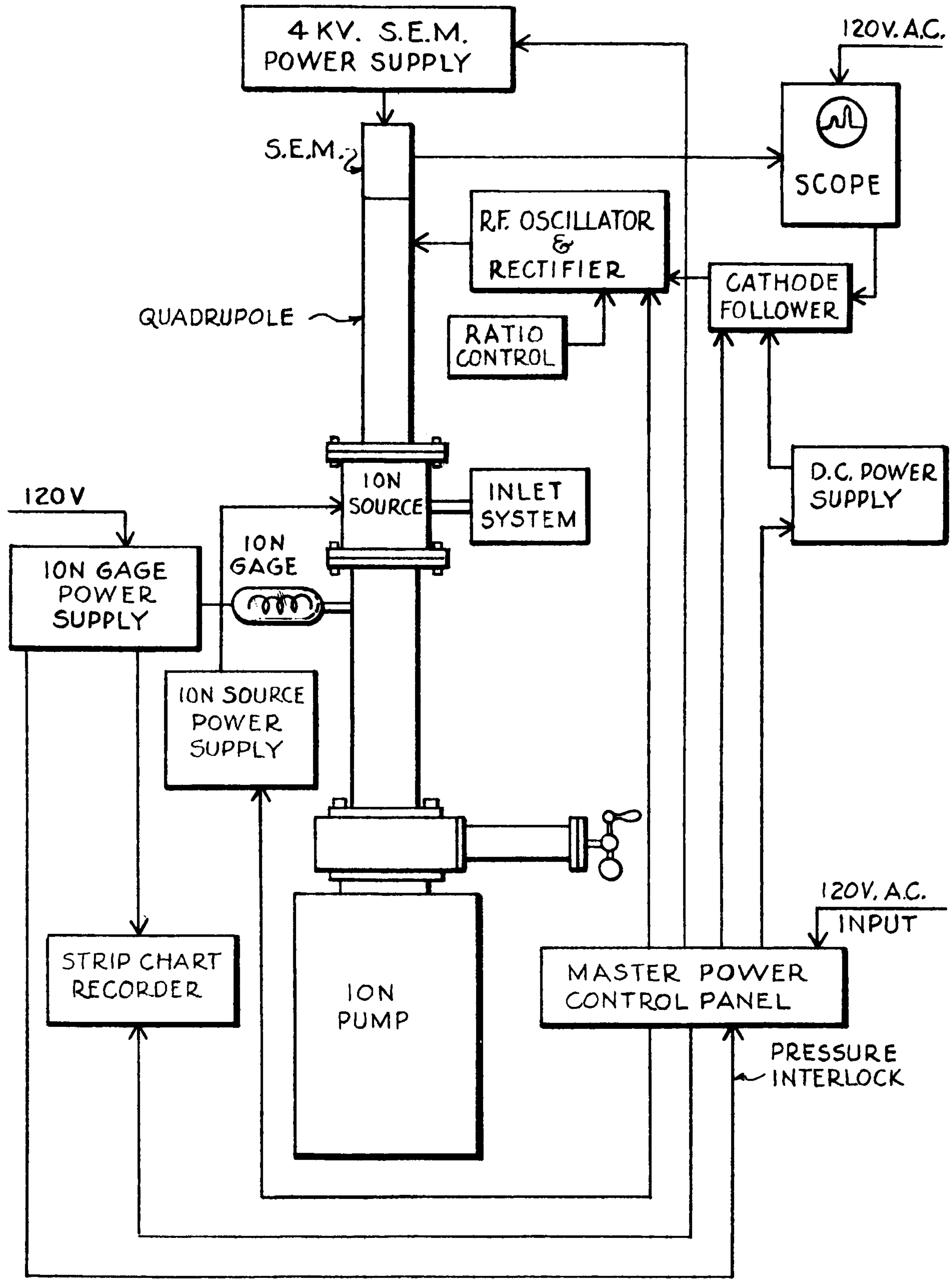


Figure 3. Apparatus Block Diagram

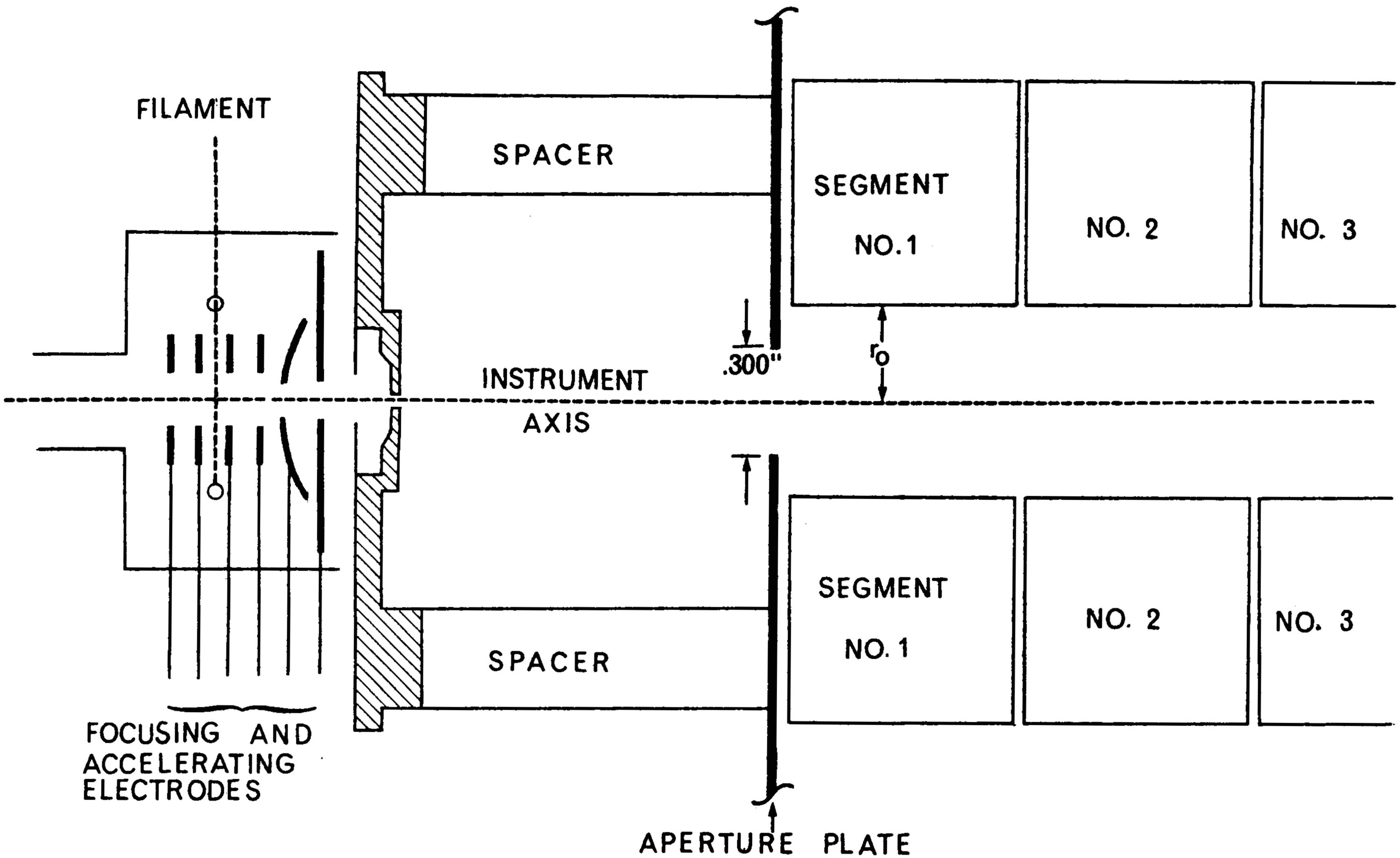


FIGURE 4

ION SOURCE & ENTRANCE GEOMETRY

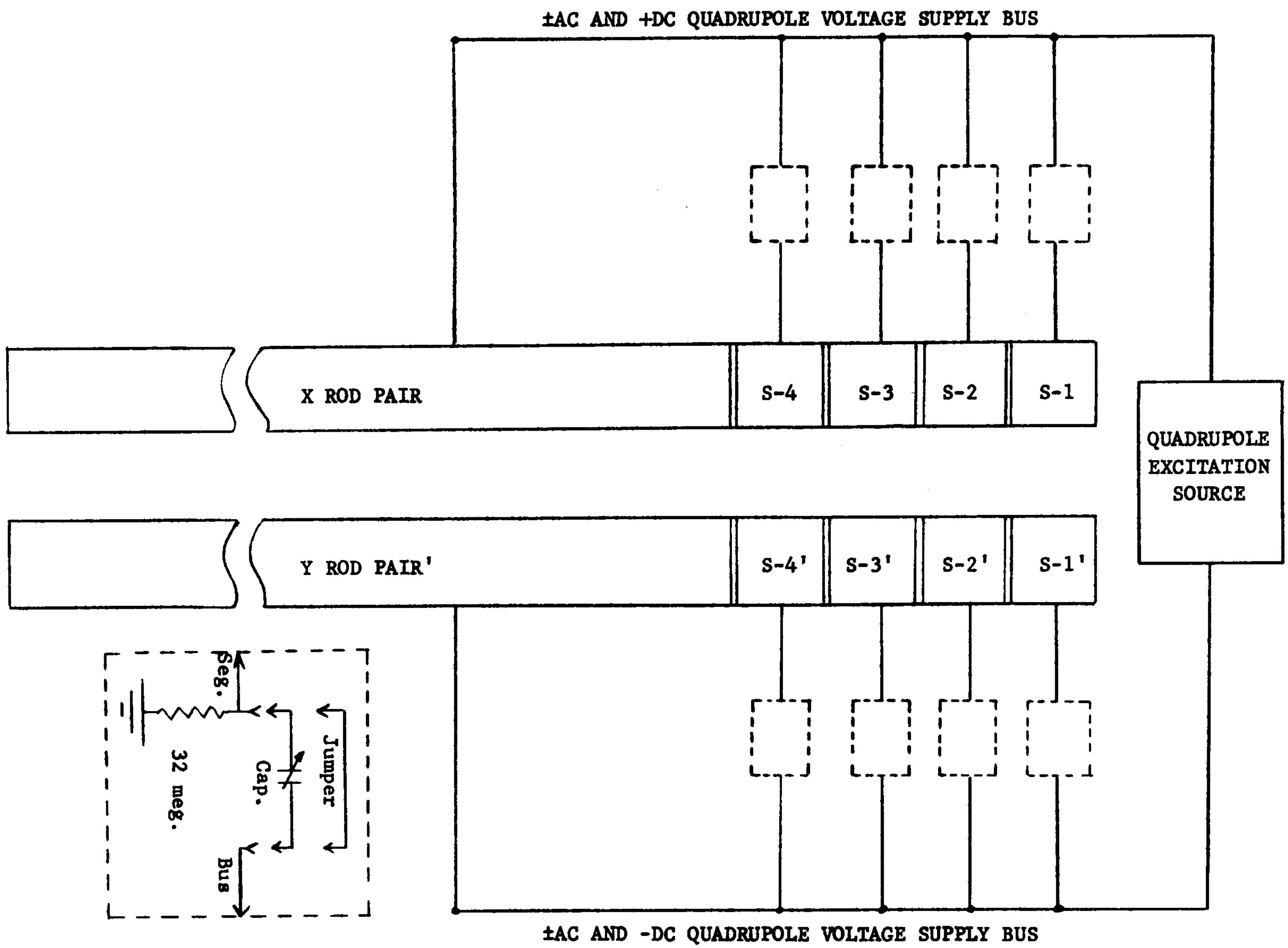


Figure 5. External Electrical Connections to Quadrupole Rod Segments

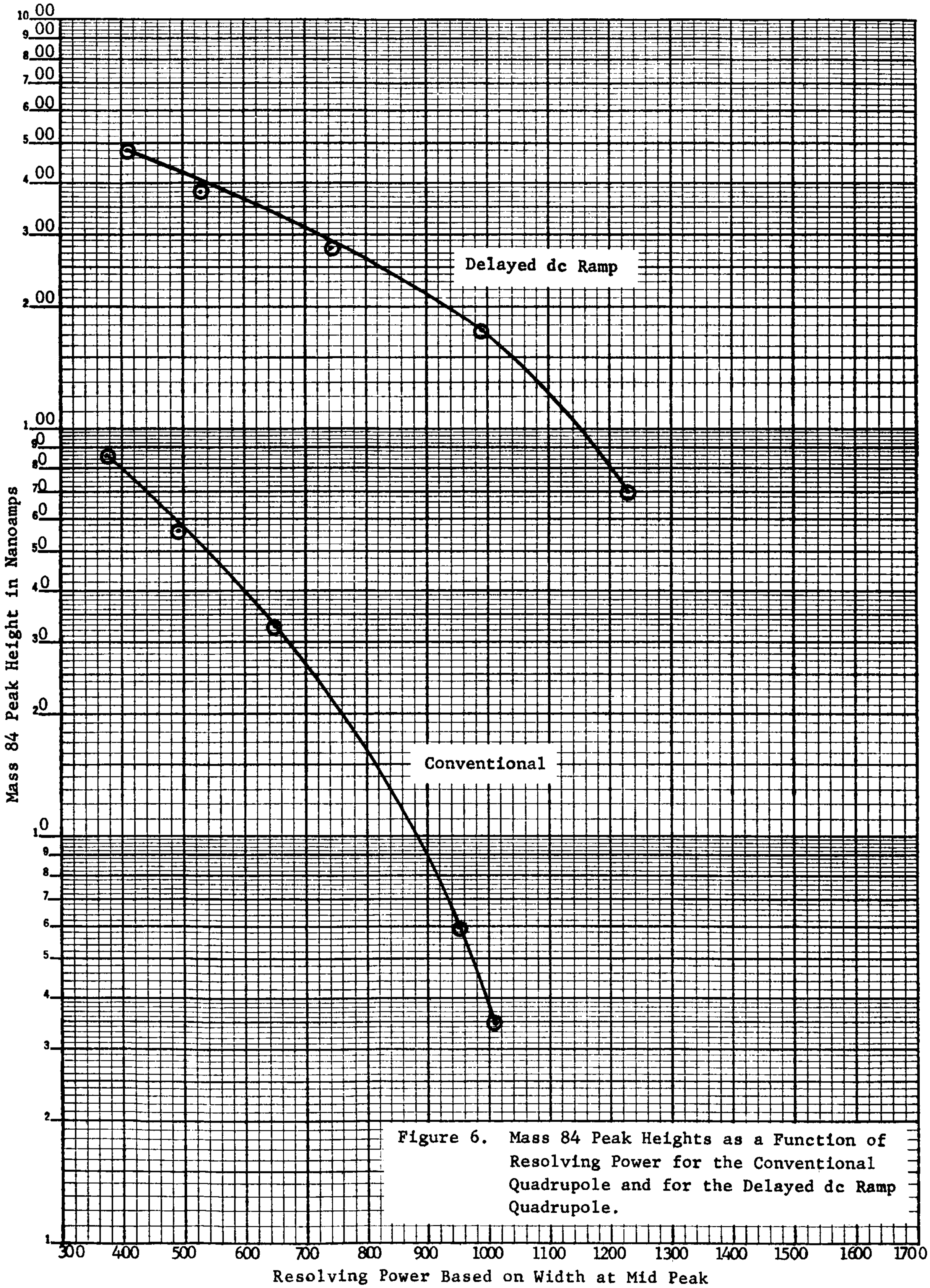


Figure 6. Mass 84 Peak Heights as a Function of Resolving Power for the Conventional Quadrupole and for the Delayed dc Ramp Quadrupole.

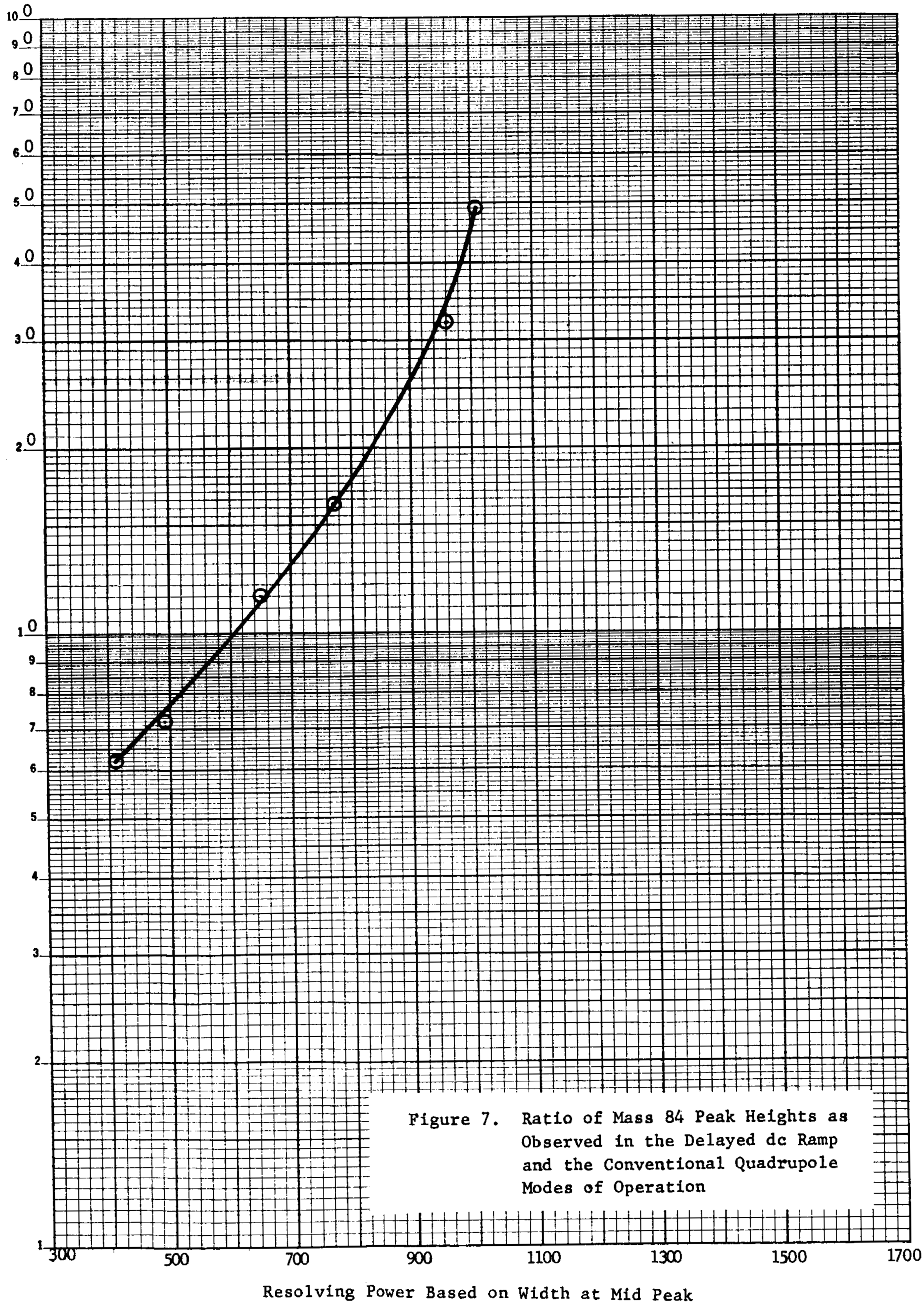
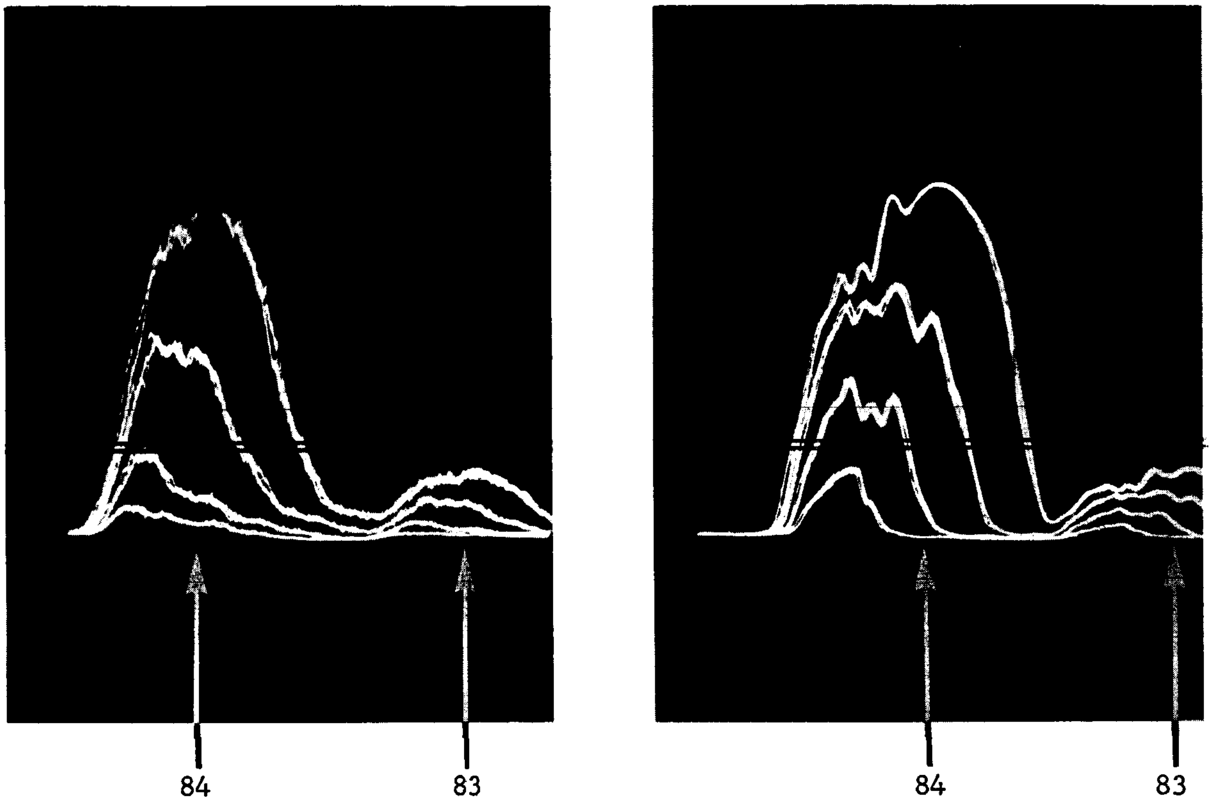


Figure 7. Ratio of Mass 84 Peak Heights as Observed in the Delayed dc Ramp and the Conventional Quadrupole Modes of Operation



Conventional Quadrupole at Zero Attenuation

Delayed dc Ramp Quadrupole at Ten Times Attenuation

Figure 8. Mass 84 and Part of Mass 83 Peaks at Different Resolving Powers for the Conventional and the Delayed dc Ramp Modes of Operation