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NATIONAL BUREAU OF STANDARDS REPORT

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A NEW RADIO FREQUENCY TIME-OF-FLIGHT MASS SPECTROMETER

By

R. M. Mills



U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

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ABSTRACT

A new type of time-of-flight mass spectrometer has been developed which, unlike most other such instruments, utilizes a continuous, nonpulsed stream of ions. The ions accelerate through a constant d-c voltage and then pass into a drift tube. A radio frequency voltage is applied at the exit and entrance gaps of the drift tube in such a way that the field at the entrance is the negative of that at the exit. Ions which travel through the drift tube in any whole number of rf cycles therefore experience no change in energy from the rf field, while all other ions experience some change. The number of ions which have not had their energy changed is measured by placing an electric barrier in front of the collector and differentiating the cut-off curve. The instrument has resolved the isotopes of rubidium at masses 85 and 87, and has identified homogenous ion beams ranging in mass from helium to cesium. Due to the nature of the instrument's output, it will be most useful in applications requiring the analysis of a simple mass spectrum, that is, spectra containing only a moderate number of different masses.

1. Introduction

This paper describes a new type of time-of-flight mass spectrometer being developed for use in combustion research. It was designed to be capable of using a flame as the ion source. Since only a limited number of ions can be withdrawn from a flame, adequate sensitivity while keeping the needed resolution has been one of the major problems of previous workers in this field [1]. The mass spectrometer has a high duty cycle and a large transmission coefficient along with moderate resolving power. However, due to the nature of the instrument's output, it will be most useful in applications requiring the analysis of a simple mass spectrum, that is, spectra containing only a moderate number of different masses.

2. Theory of Operation

In general, time-of-flight (T.O.F.) mass spectrometers depend on the fact that heavier ions take a longer time to traverse a given distance than do light ions. The velocities of the ions vary according to:

$$v = \sqrt{\frac{2eV}{m}} \quad (1)$$

where e is the charge of the ions, V is the total voltage through which the ions have been accelerated, and m is the mass of the ion. If a pulse of ions of various masses enters a field-free drift tube, after being accelerated, the ions will travel in bunches corresponding to their mass. The distance between bunches will increase the further the ions travel down the drift tube. The mass spectrum can then be measured by observing the ion current collected at the exit end of the drift tube as a function of time. In some instruments, electrical gates set for the proper delay are used in front of the collector to improve the resolution. However, all instruments of this type have to be pulsed at the source and the time between pulses must be great enough to clear the drift tube of ions from the previous pulse. The pulsing greatly reduces the duty cycle of the mass spectrometer which effectively lowers its sensitivity. A T.O.F. mass spectrometer has been developed which appears to be less subject to this defect. It operates by accumulating the ions continuously and concentrating them between each pulse [2]. However, besides adding to the complexity of the instrument, this introduces an uncertainty about recombinations and other ionic changes during the delay between pulses.

Another type of T.O.F. instrument which is described in the literature [3,4] distinguishes masses by the energies the ions receive from a series of rf fields. A continuous beam of ions pass through 10 or more electrodes which are perpendicular to the beam path. The geometry of these electrodes and frequency of the rf fields maintained between them are such that ions of one mass receive more energy than the others. An electrical barrier is placed in front of the ion collector. Only ions which have received extra energy from the rf fields are able to overcome the barrier and reach the collector. This instrument gives good resolution, but has several drawbacks. The spacing between the electrodes is critical for proper operation which makes construction and modification difficult. The numerous electrodes

through which the ions must travel reduce the transmission of the instrument. And finally, this type of instrument collects pulses of ions even though a continuous beam of ions enters the analyzing region. This effect is due to the fact that an ion of correct mass must also have the correct initial phase with respect to the rf voltages as it enters the fields in order to gain the necessary amount of energy to reach the collector. Thus the sensitivity of that instrument is reduced in the same way as with the pulsed type of spectrometer.

The mass spectrometer reported here resembles the last instrument described in that ions of different masses are distinguished by their energies rather than their special positions; it differs in that there are no initial phase requirements. Consider a drift tube of length L which has rf electric fields applied at its ends as shown in Figure 1. It is seen that the field applied at one end is the negative of the field applied at the other end. The rf waves are represented in Figure 2. Suppose an ion enters the drift tube at some time A_1 . If the frequency of the rf voltage and the velocity of the ion are such that the ion emerges at the other end of the drift tube after one period of the rf wave, the ion will pass through the second rf field at time A_2 . It is seen that no matter what part of the cycle the ion enters the drift tube, the energy which it obtains at the first field is exactly balanced out by the energy it loses at the second. Consider now, a heavier ion which takes a longer time to traverse the drift tube. For example, if it enters the drift tube at time B_1 , it might pass through the exit field at time B_2 . In general, ions which stay in the drift tube a complete cycle or any multiple, n , of complete cycles neither gain nor lose energy. These are the ions which cause the detection system to give output signal.

3. The Detection System

As stated before, the detection system must identify the resonant ions by their energy. This measurement is made by retarding field method [5,6]. An electrical barrier is placed immediately in front of the Faraday cage collector. For a given barrier potential, E , measured with respect to the ion sources, only ions with energies equal to E electron volts or greater can pass. Or

$$n(E) = \int_E^{\infty} N(e) de \quad (2)$$

where $n(E)$ is the number of ions per unit of time that penetrate a barrier of height E and $N(e)$ is the number of ions approaching the barrier per unit of time with energy e at the barrier. $N(e)$ is obtained from equation (2) by differentiating with respect to E /

$$\frac{dn(E)}{dE} = -N(e) \text{ at the point where } e = E. \quad (3)$$

Since the detection system must measure the number of ions which have had their energies unchanged by the rf fields, $N(0)$ is the quantity sought. Figure 3 is a set of cut-off curves showing collector current as a function of barrier height in volts, obtained with an x-y recorder. The center curve is the cut-off curve for an ion beam homogeneous in mass which is in resonance with the rf voltage, taken with n equal to 12. The other curves were taken with n equal to 11.5, 11.7, 12.2, 12.4.

The differentiation represented in equation (3) is performed by introducing a variation in E about the point $E = 0$ shown as ΔE in figure 3. The resulting a-c component in the collector current is proportional to $dn(E)$. And $dn(E)$ is proportional to $N(e)$ if dE is infinitely small. The equipment seems to give best results using a variation of 10 volts (i.e. $\Delta E = 5$ volts) and this value will be assumed in the remainder of this paper. Using a dE of finite size means that an average value of N is actually measured over the range $E = 0 \pm 5$ volts.

If the ion beam is not homogeneous in mass, the resulting a-c signal is the sum of the contributions from each mass. The variation in the ion current is amplified by a-c amplifiers and then fed to a phase detector, ΔE supplying the reference phase used in the detector. An x-y recorder is connected to the output of the detector. The masses are swept by changing the frequency of the rf voltage. A potentiometer is attached to the tuning knob of the rf generator. The voltage from the potentiometer is thus directly related to the rf frequency and is connected to the other input of the x-y recorder. The potentiometer and tuning knob are driven by a one rpm motor. No development work has been done to make this instrument a fast-sweep instrument, capable of displaying its output on an oscilloscope, but this seems feasible.

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For the rules in differentiating under the integral sign, see any text such as Hilderbrand's "Advanced Calculus for Engineers," p. 352.

4. Resolution

An indication of the resolution which can be expected with this instrument can be obtained by writing equation (1) with $v = L/t$:

$$t = L \sqrt{\frac{m}{2eV}} \quad (4)$$

where t is the time-of-flight of the ion through the drift tube and L is the length of the drift tube. Differentiating:

$$dt = \frac{\partial t}{\partial m} dm + \frac{\partial t}{\partial V} dV = 1/2 \frac{t}{m} dm - 1/2 \frac{t}{V} dV \quad (5)$$

Assume for the present that V_{rf} is small enough compared to the d-c accelerating voltage so that the second term in equation (5) can be neglected. Then equation (5) can be written as:

$$\frac{dt}{dm} = \frac{t}{2m} \quad (6)$$

This expression gives the difference in flight time between ions of mass m and $m + dm$. An expression for the net change in voltage or energy, dX , which an ion receives from the rf voltage is obtained by multiplying equation (6) by the time rate of change (or slope) of the rf voltage:

$$\frac{dX}{dm} = \frac{dt}{dm} \frac{dV_{rf}}{dt}$$

Since the rf voltage is a sine wave, the magnitude and sign of the slope is a function of time. The resolution depends on the magnitude of the slope, but is independent of the sign. Therefore, equation (6) is multiplied by the rms value of the slope, equal to $\frac{\pi V_{rf}}{\sqrt{2} T}$. V_{rf} is defined as the peak to peak voltage of the

rf wave and T as its period: $\frac{dX}{dm} = \frac{\pi t V_{rf}}{\sqrt{2} 2m T} \quad (7)$

If m is the resonant mass, the above expression gives the energy, in electron volts, which an ion different in mass from m by dm receives from the rf field. Since $t = nT$, equation (7) can be written as:

$$\frac{dX}{dm} = \frac{\pi n V_{rf}}{2\sqrt{2} m} \quad (8)$$

It is interesting to note that had the rf voltage been a saw-tooth wave with constant slope (that is, rising linearly with time to V_{rf} and then falling quickly back to zero) equation (6) would have been multiplied by V_{rf}/T , making equation (8) equal to $\frac{nV_{rf}}{2m}$.

Therefore, better resolution should be obtained using a sine wave rather than a saw-tooth wave, and this was verified experimentally.

Equation (8) indicates that the resolution increases with V_{rf} . However, if V_{rf} is too large compared to the d-c voltage through which the ions have been accelerated, the last term in equation (5) becomes significant. This means that the rf voltage begins to modulate the energy and thus the flight time of the ions through the drift tube. It is desirable, however, that the variation in flight time expressed in equation (5) be due mostly to mass difference rather than voltage fluctuations. Experiments indicate that best results are obtained in separating masses m and $m + dm$ when

$$\frac{V_{rf}}{V_{dc}} \approx 2 \frac{dm}{M} \quad (9)$$

A second condition for good resolution is that V_{rf} be at least several times as great as the variation in ΔE .

Equation (8) also shows that the resolution increases with n . Figure 4 shows the peaks of K^{39} and K^{41} for values of n equal to 8 through 20 (i.e., the 8th through 20th harmonics). It can be seen that the separation of the isotopes does increase for higher values of n . Two conditions place an upper limit on the value of n . Since for a given mass, n is proportional to the frequency, the usual engineering problems of high frequency can become troublesome. The other limitation results from a decrease in spacing between harmonics for higher values of n . When optimum values of n and V_{rf} are used, the maximum resolution that can be obtained from this instrument depends on how small a change in ion energy, dX , can be detected. This, in turn, depends on how monoenergetic the ions are to begin with at the source, how monoenergetic they remain as they enter and travel down the drift tube, how well focused the beam is at the barrier [7], and how sharply the barrier and collector can cut off ions with insufficient energy.^{2/}

2/

For sharp cut off, the barrier must be such that no field can penetrate through. The collector must be able to collect ions of large and small energy with equal efficiency and be able to prevent ions which have experienced elastic reflections from escaping the collector.

5. Harmonics

As has been mentioned before, ions may stay in the drift tube any whole number, n , of cycles in order to be detected. Thus, as the frequency is varied, the mass peaks will repeat corresponding to various values of n as in Figure 4. The two rf frequencies at which two adjacent harmonics of a particular ion appear must be measured to determine the ion's mass. The ratio of these two frequencies is equal to the ratio of their harmonic numbers. That is:

$$\frac{f_n}{f_{n+1}} = \frac{n}{n+1} \quad (10)$$

This equation can be solved for n , or n can be found conveniently on a slide rule. The procedure is to look for two consecutive integers which have the same ratio as f_n/f_{n+1} . Knowing n and the corresponding frequency, f_n (equal to $1/T_n$), t is found from $t = nT_n$, which in turn gives m in equation (4).

The determination of m becomes difficult if the ion beam contains ions of many masses. The harmonics of the mass peaks become too cluttered to pick out two adjacent harmonics. In applications requiring the study of complex beams, it would be desirable to eliminate all but one of the harmonics for each mass. Partial elimination of the harmonics has been achieved by applying two rf frequencies referred to as f_1 and f_h as in Figure 5. Frequency f_1 is generated from f_h by frequency division so that the ratio of f_h to f_1 is known whole number, say r . With the present equipment, r can be chosen to be equal to 2, 4, 8, 16 or 32. In order for the ions to escape a change in energy, they must pass through two drift tubes, emerging from each tube at the same rf phase that they entered. Let n_1 be the number of rf cycles an ion takes to travel through the first drift tube, and n_h the number of rf cycles an ion takes to travel through the other tube. Since both tubes are of equal length, the transit times for both tubes must be equal. That is, $n_1 T_1 = n_h T_h$. By definition, $f_1 = 1/T_1$ and $f_h = 1/T_h$. Thus, if n_1 is equal to one, n_h must be equal to r . An ion which would pass through the second drift tube with $n_h \neq r$ does not pass through the first drift tube in a whole number of rf cycles. The voltage at frequency f_1 discriminates against the unwanted harmonics while the voltage at frequency f_h resolves the masses as before. As was the case in using a single drift tube, the rf

voltages can be increased until the improvement in resolution is balanced out by the adverse effects of velocity modulation. The value for the optimum rf is again expressed in equation (9). Had the two voltages been applied together to one long drift tube, the sum of their magnitudes would have to be less than $2V_{dc} \frac{dm}{m}$. Applying them separately, each rf voltage must be less than $2V_{dc} \frac{dm}{m}$, resulting in larger permissible magnitudes.

Figure 6 was obtained by using this principle of harmonic elimination. It shows the frequency range where the 10th through 28th harmonics of potassium would normally appear. The largest peak at the center is the 16th harmonic of K^{39} . The two small peaks on either side are the 15th and 17th harmonics of K^{39} . Figure 6 was made using the maximum low frequency voltage which the equipment could generate. It is felt that better results could be obtained by increasing this voltage, but up to now, no further developmental work has been done along this line.

6. Results

The following positive ions have been detected with this instrument: ^{39}K , ^{41}K , ^{23}Na , 4He , N_2 , N , ^{35}Cl , ^{37}Cl , ^{85}Rb , ^{87}Rb , ^{133}Cs . All except the helium and nitrogen ions were obtained by placing salts on a hot tungsten wire filament [8]. Helium and nitrogen were ionized by heating a bare tungsten filament in atmospheres of helium and nitrogen at reduced pressures.

A calibration factor defined as:

$$CF = \frac{1}{T_n} \frac{\sqrt{m}}{n} = \frac{\sqrt{2eV}}{L} \quad (11)$$

was measured for each of the ions listed above. If m is in atomic mass units ($Q^{16} = 16.00$) and $1/T_n$ in megacycles, the calibration factor was found to be:

$$CF = 0.678 \pm 0.003 (\approx \pm 0.4\%)$$

This figure was obtained by using known values of mass for the above ions. The frequencies at which the various harmonics appeared were measured with a timer-counter. The d-c accelerating voltage

was approximately 3,000 volts, and the drift tube length was roughly 1.12 meters long (0.56 meters long when used with the harmonic eliminator). With the present source, it is necessary to apply a focusing field in the region where the filament is located. The proper focusing voltage changes each time the filament is removed to replace the salt. It has been found that this change in focusing voltage produces a slight change in the calibration factor, accounting for a portion of the 0.4% variation. For a fixed value of the focusing voltage, the calibration factor is found to vary about 0.1%. Figures 7a and 7b show the 30th through 48th harmonics of Rb^{85} and Rb^{87} (a), and the 15th through 37th harmonics of Cs^{133} (b). Figure 8 is a mass spectrum showing K^{39} , K^{41} , Cl^{35} and Cl^{37} . Some variation in the peak heights of the harmonics can be observed in figures 7b and 8. Duplicating these graphs shows that this fluctuation is due to the instability of the source. Little care has been taken, up to date, to ensure linearity in peak heights. In Figure 9, the ion source at the right, the tube containing the drift tube, the collector shielding box, and some of the electronic equipment are visible.

7. Acknowledgments

The author wishes to thank Mr. Carroll E. Creitz for his help and interest, Mr. William H. Bailey for his shop work, Mr. Louis A. Marzetta and Mr. Malcom S. Morse for their work in developing the rf generators, and others who have made helpful suggestions.

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TO ΔE VOLTAGE GENERATOR

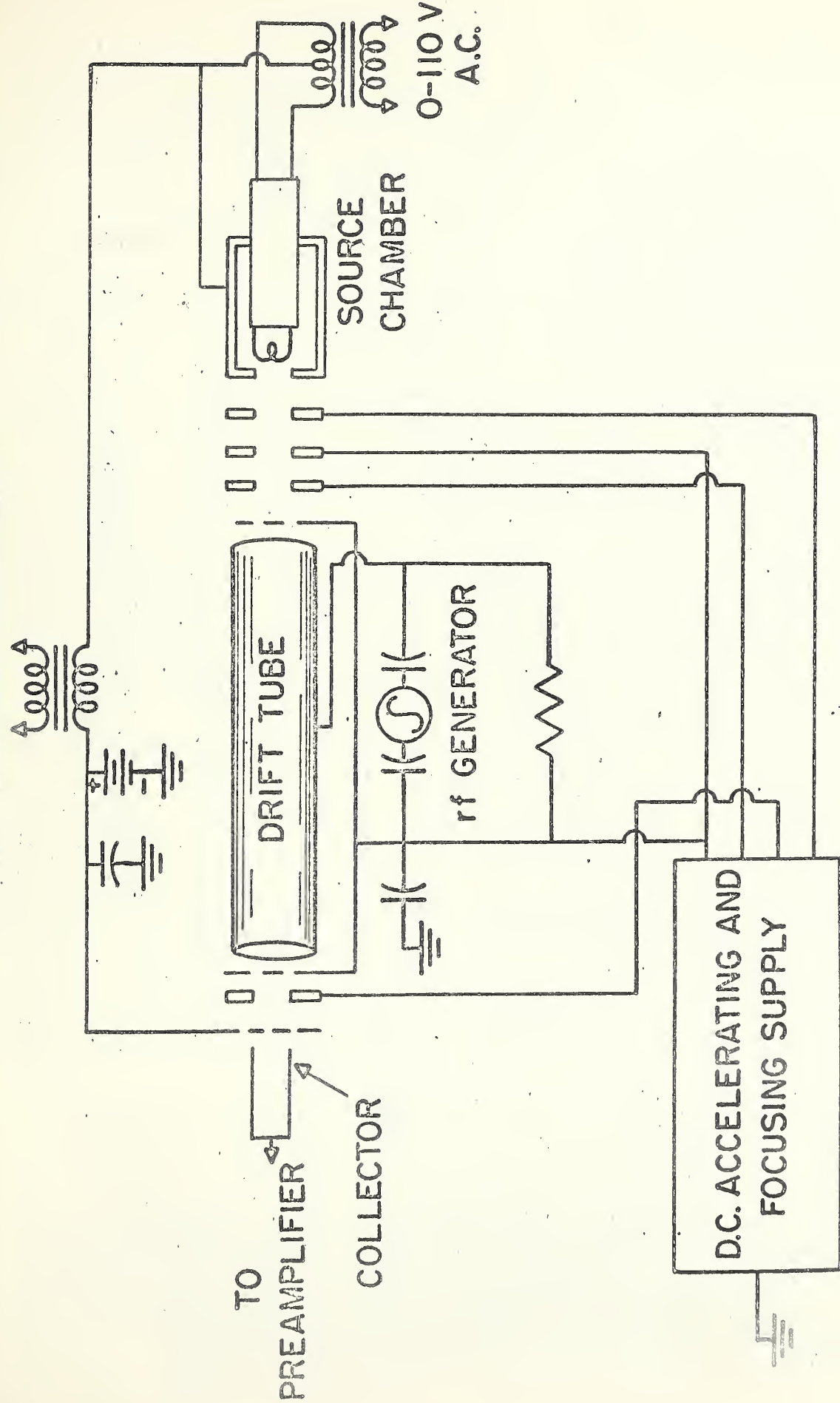


Figure 1. Simplified diagram of the Mass Spectrometer.

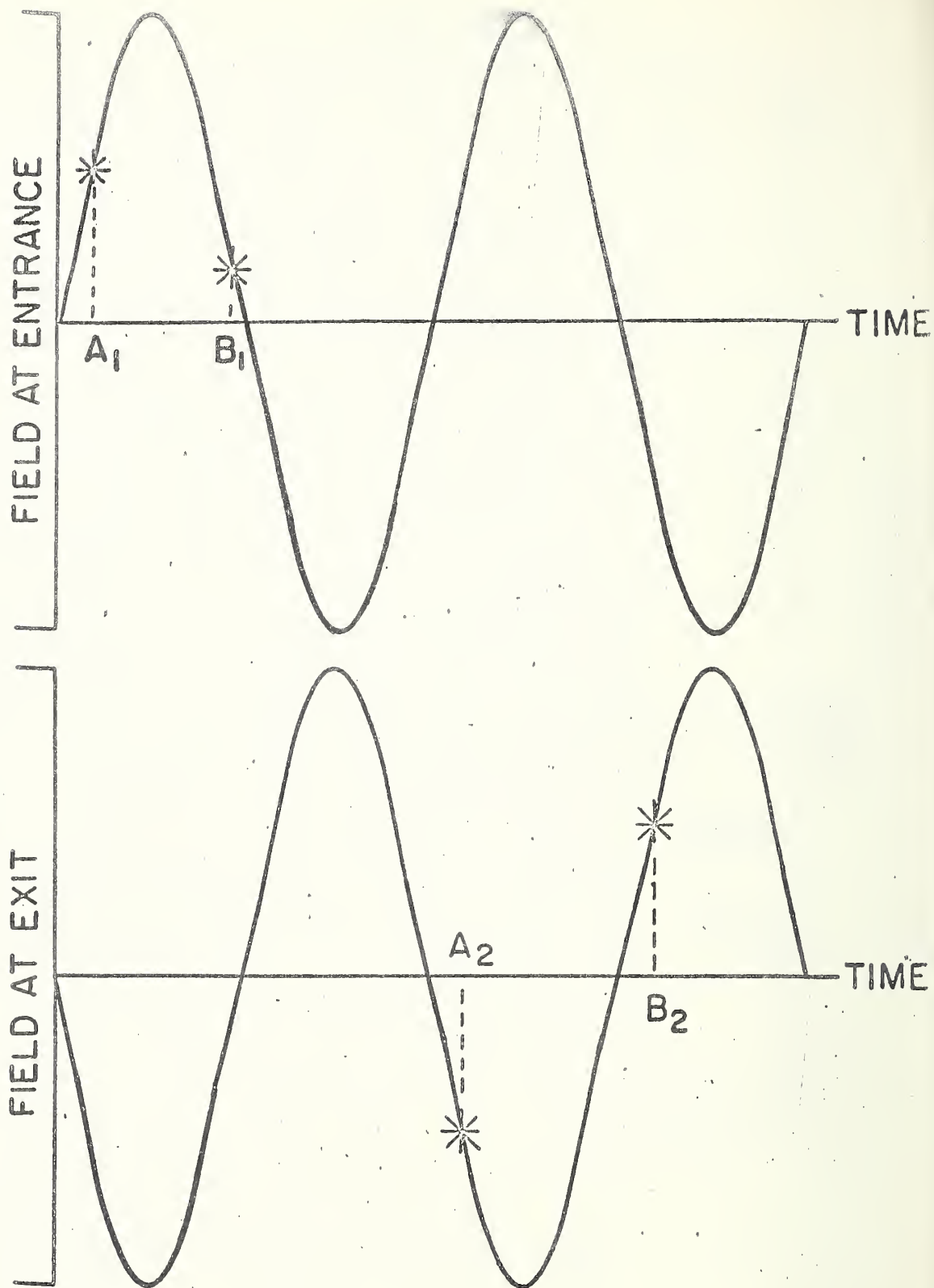


Figure 2. Radio frequency fields at entrance and exit of a drift tube. A resonant ion entering at time A_1 , and leaving at time A_2 experiences no change in energy. A typical nonresonant ion entering at time B_1 , and leaving at time B_2 experiences a net gain in energy.

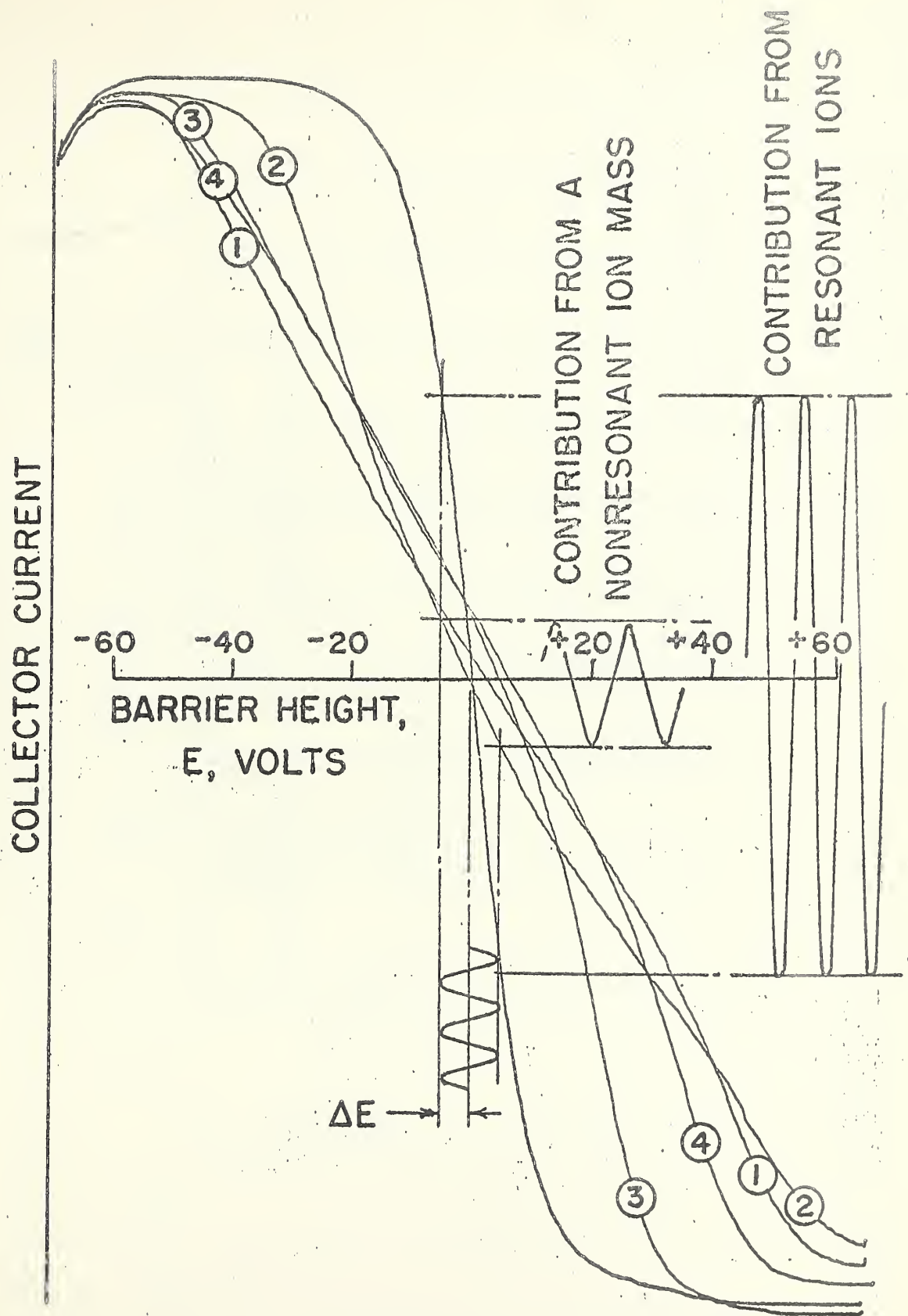


Figure 3. Cut-off curves for $n = 12.0$ (central curve) $n = 11.5$ (1), $n = 11.7$ (2), $n = 12.2$ (3), and $n = 12.4$ (4) showing the difference in output signals for resonant and nonresonant ions.

$n=8$

$n=20$

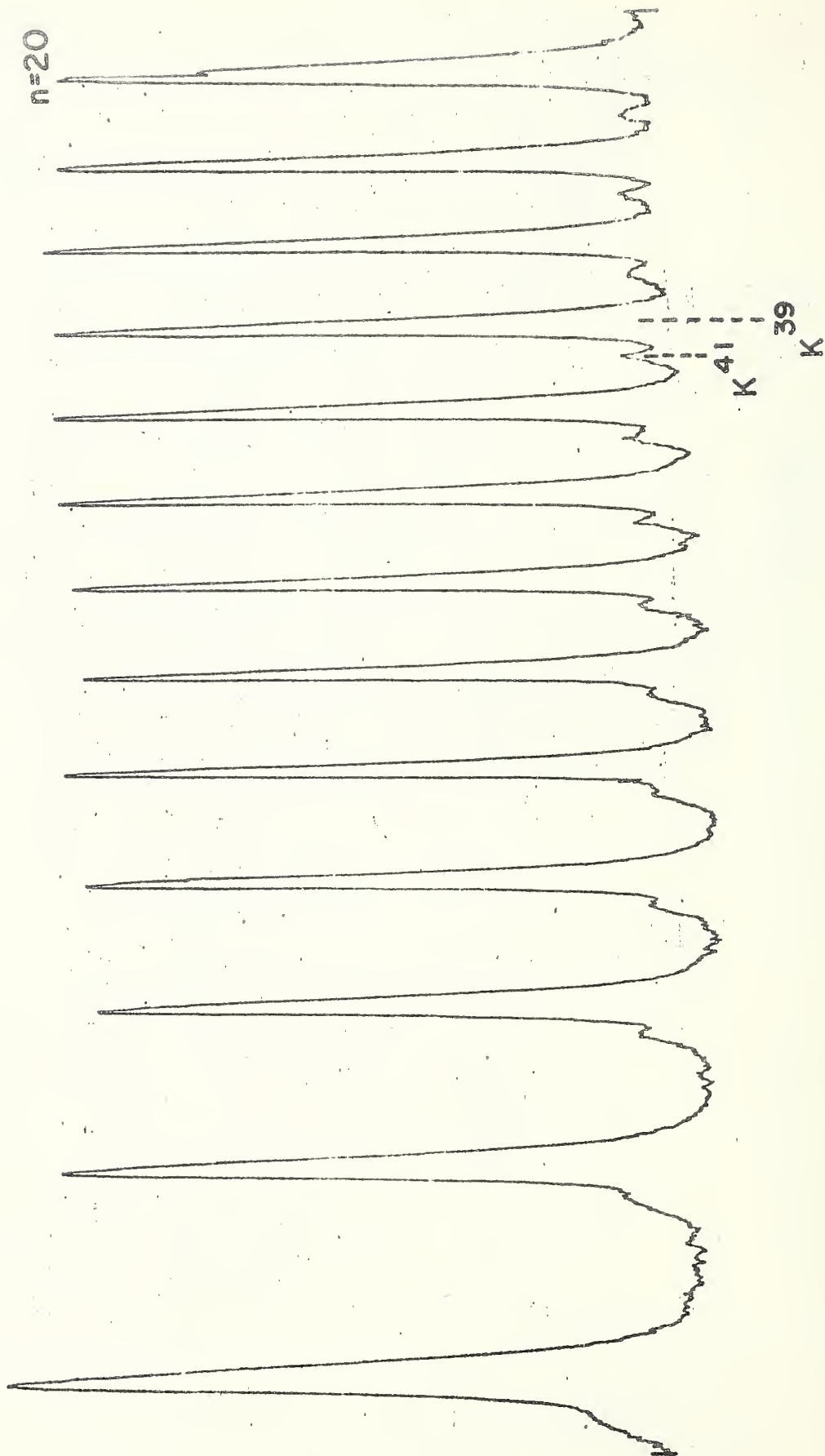


Figure 4. Eighth through twentieth harmonics of K^{39} and K^{40} .

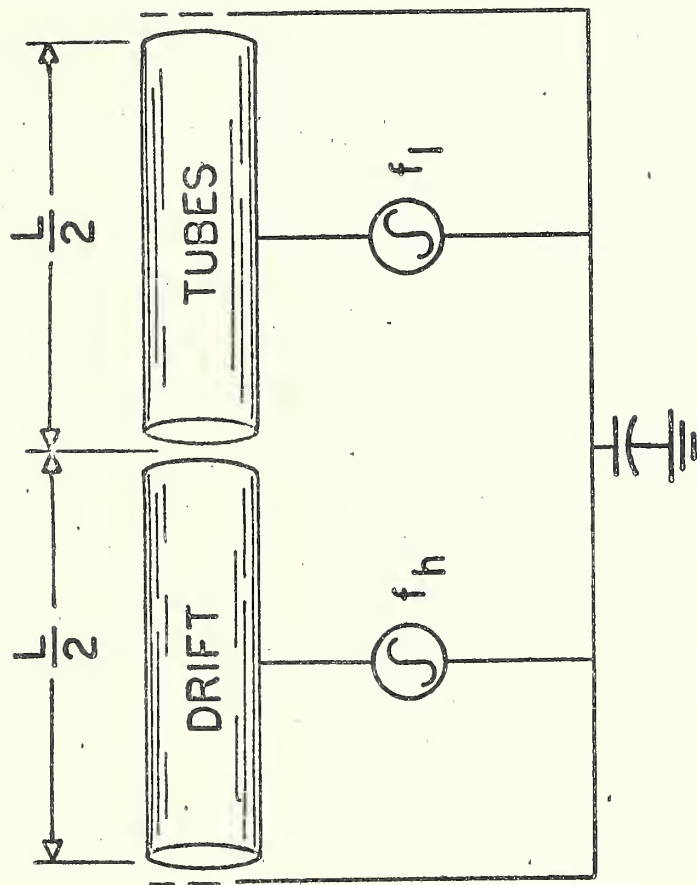


Figure 5. Drift tube arrangement used to eliminate harmonics.

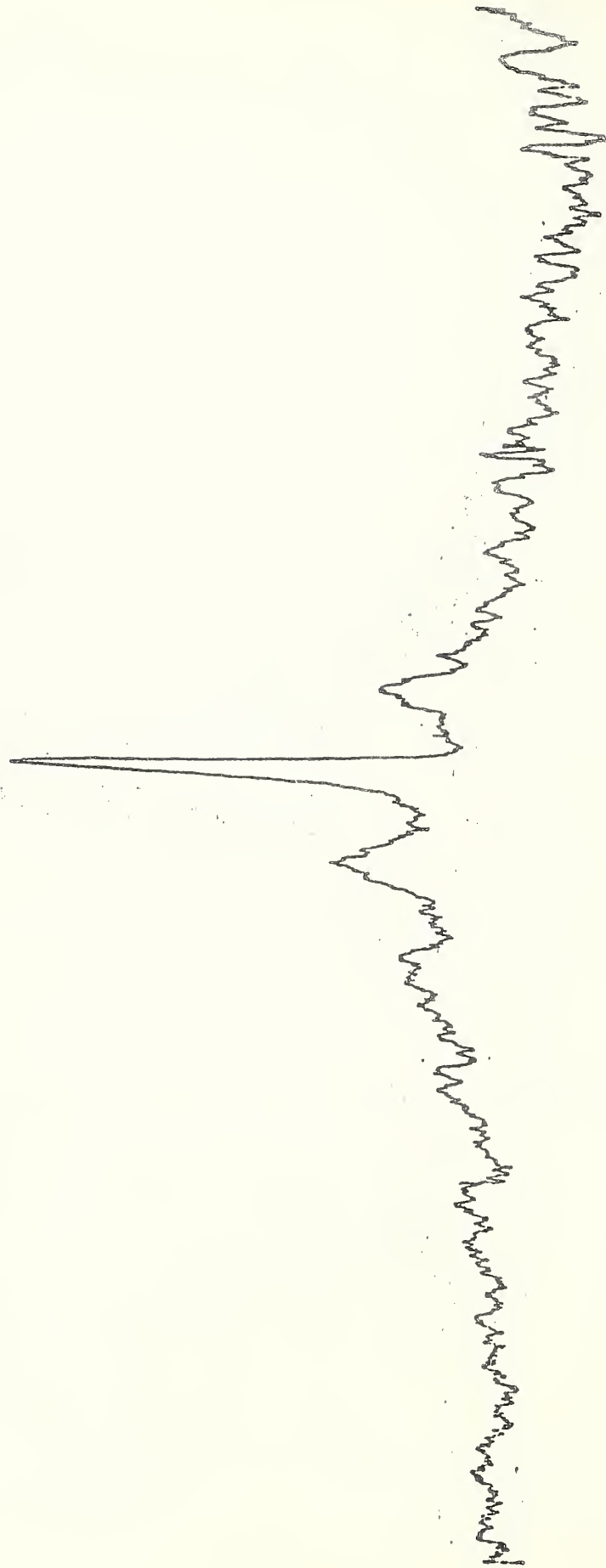


Figure 6. Mass spectrogram of K^{39} using the harmonic elimination equipment.

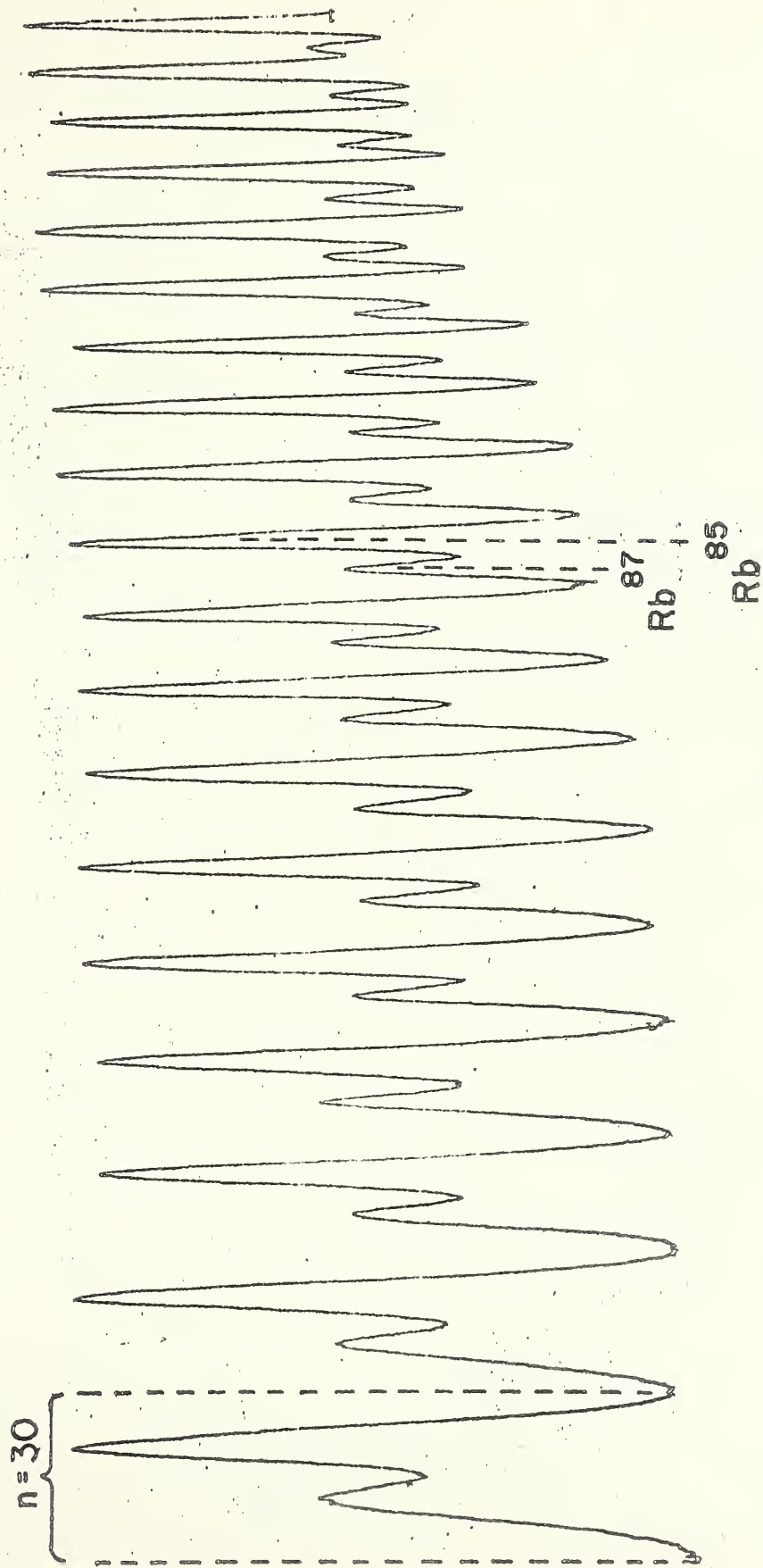


Figure 7a. Harmonics of Rb⁸⁵ and of Rb⁸⁷.

$n=15$

Figure 7b. Harmonics of Cesium.

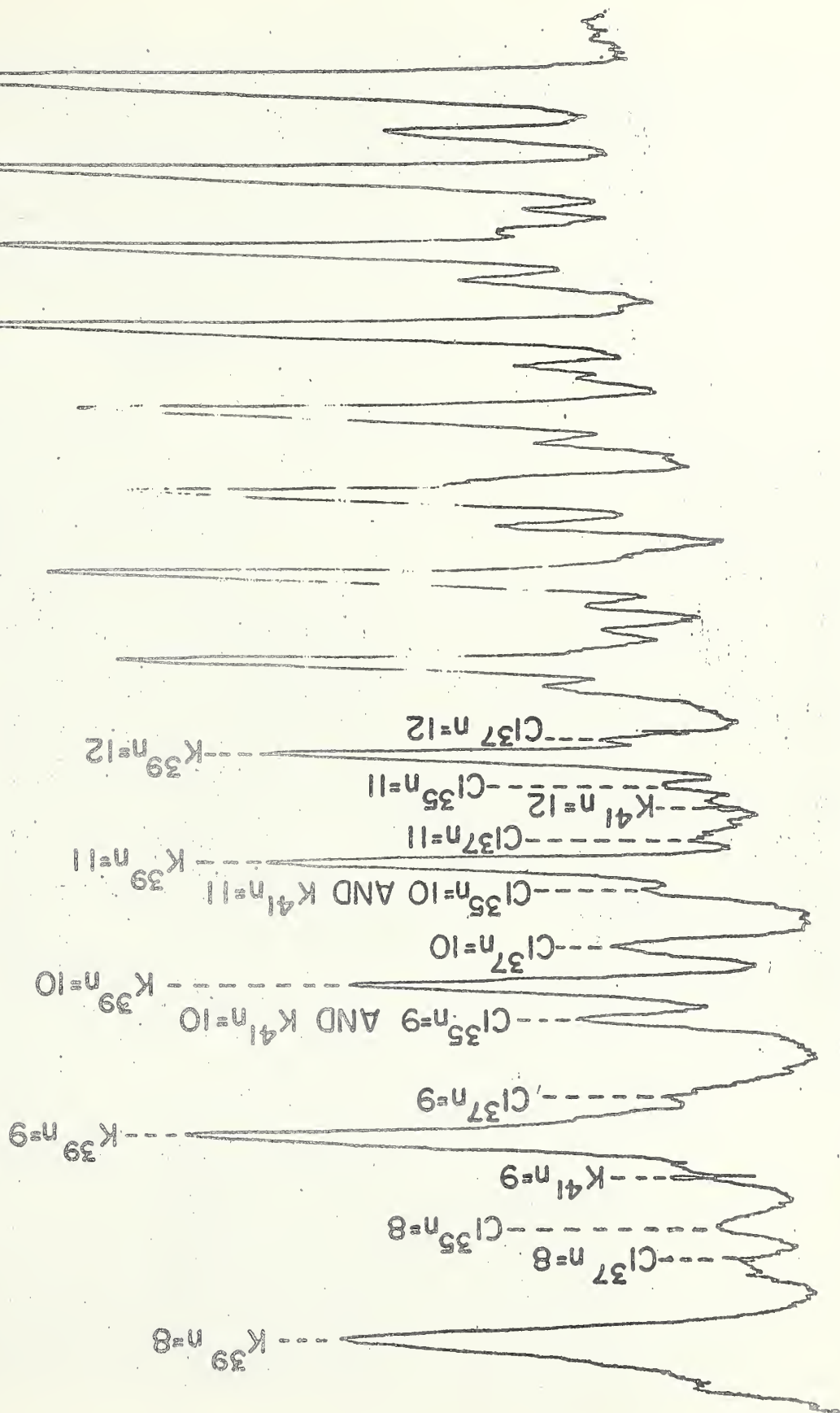


Figure 8. Harmonics of K_{39} , K_{41} , C_{135} , and C_{137} .

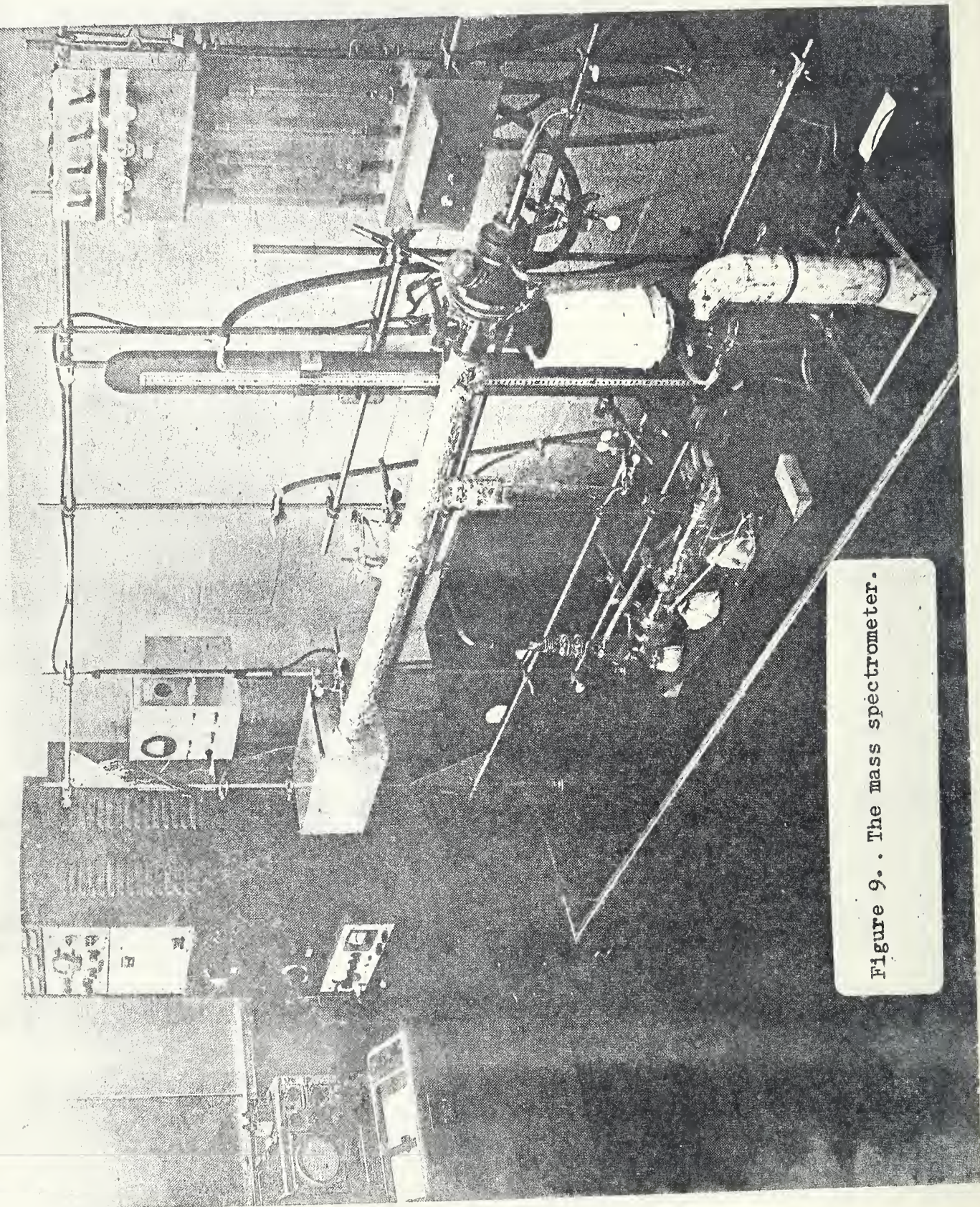


Figure 9.. The mass spectrometer.

U. S. DEPARTMENT OF COMMERCE

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THE NATIONAL BUREAU OF STANDARDS

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