Jan. 27, 1953 H. W. WASHBURN ET AL 2,627,034

MASS SPECTROMETRY

Filed March 24, 1947 2 SHEETS--SHEET 1

FIG. 1.

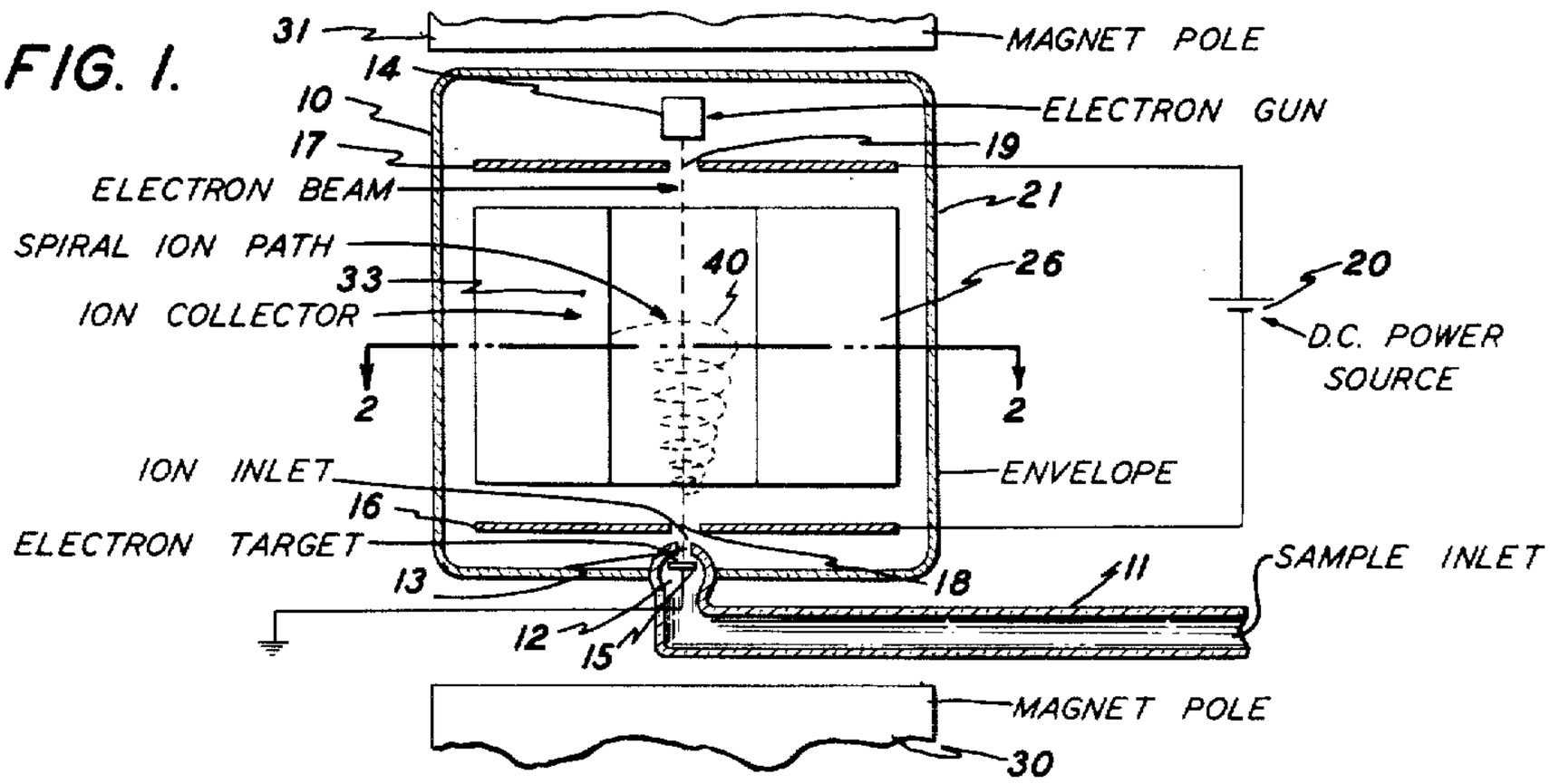

FIG. 2.

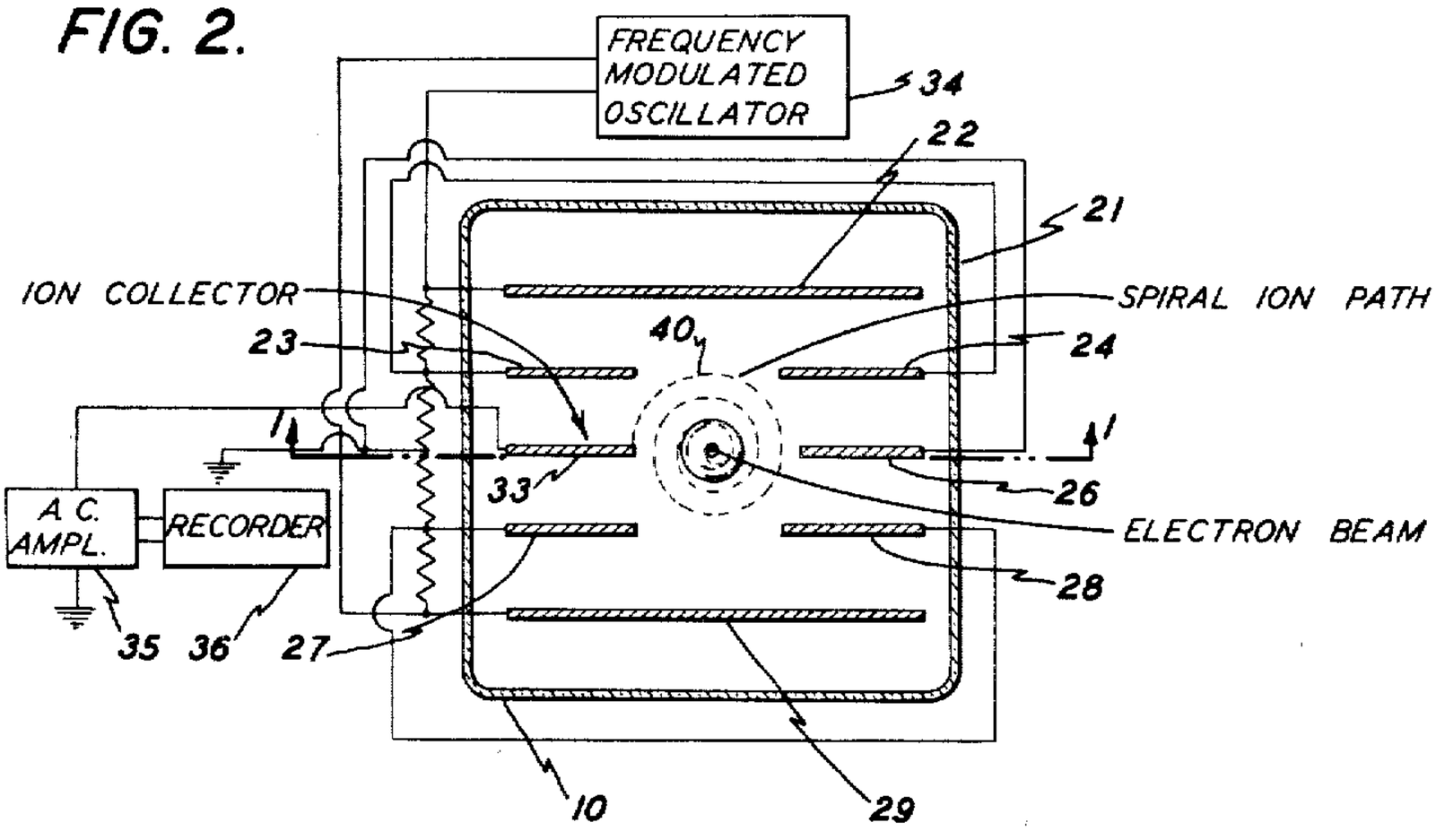

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FIG. 3.

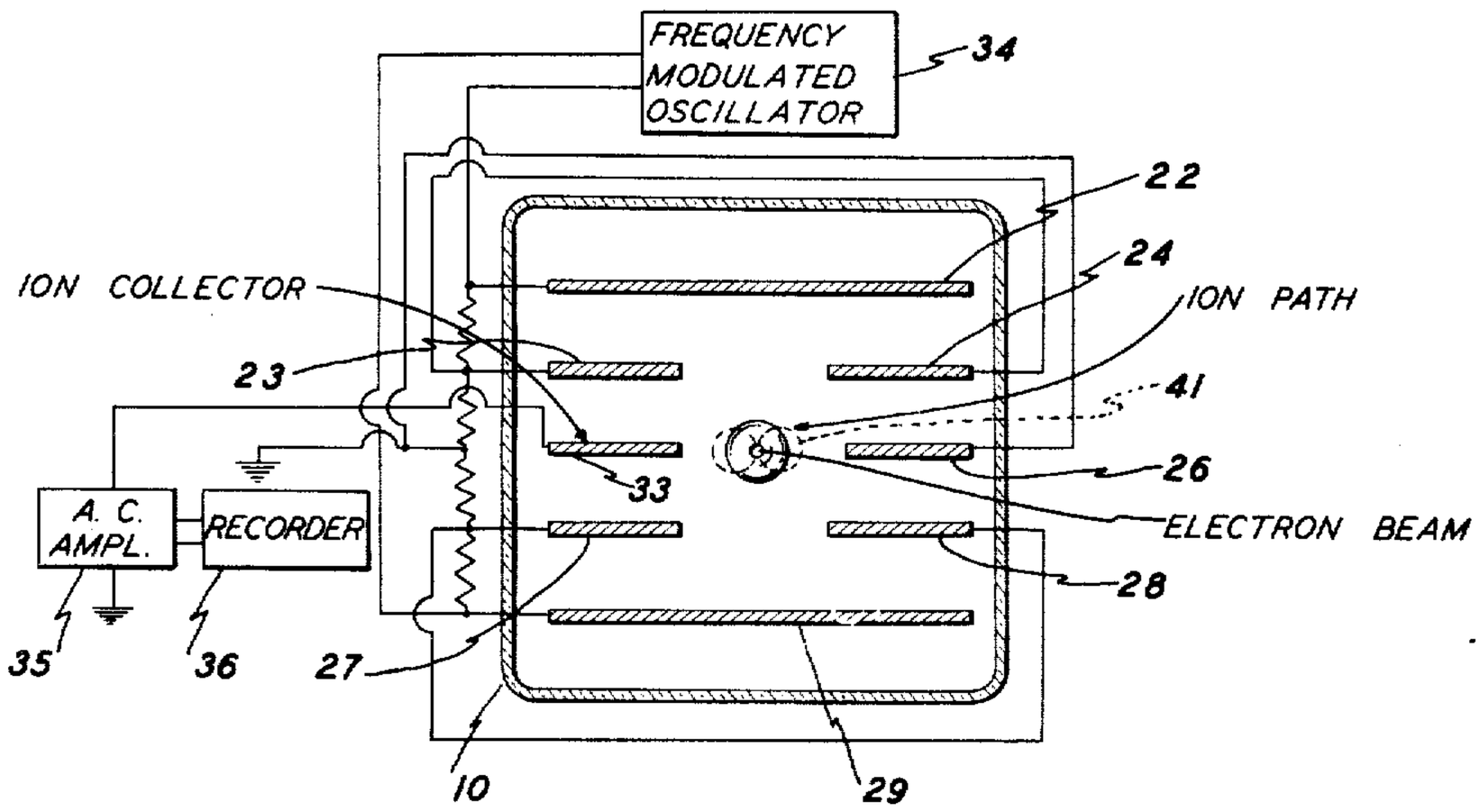
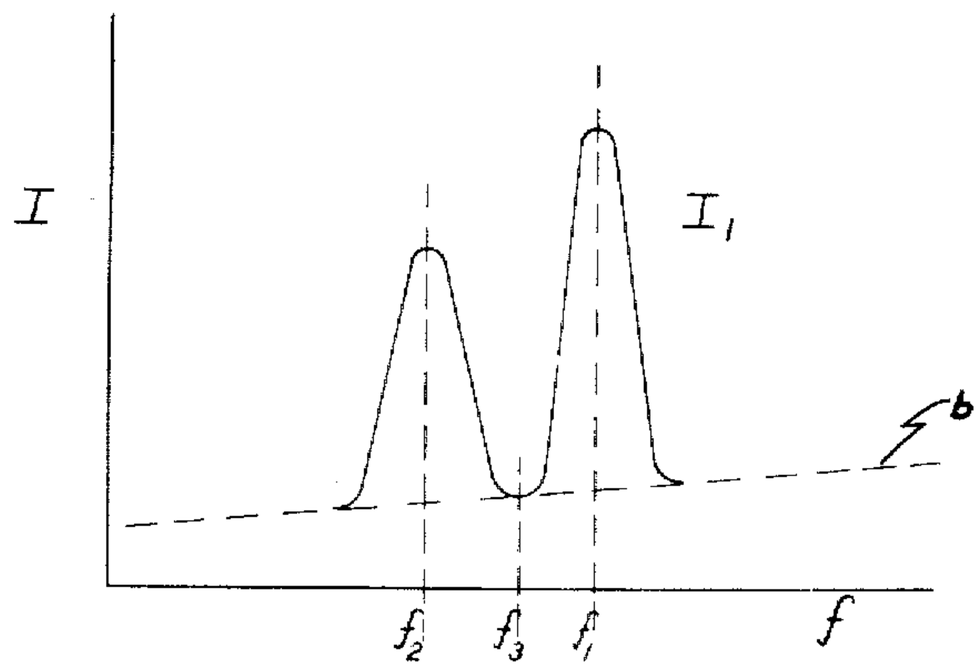


FIG. 4.



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MASS SPECTROMETRY

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Application March 24, 1947, Serial No. 736,758

22 Claims. (Cl. 250—41.9)

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This invention relates to mass spectrometry and contemplates improvement in mass spectrometry by the provision of an instrument wherein the path of the ions to be analyzed is in the nature of an expanding spiral.

In mass spectrometry, a gas sample is bombarded by moving electrons to produce ions of various substances present in the sample, and the ions thus formed are separated into various components having different mass-to-charge ratios by subjecting the ions to the influence of electric or magnetic fields or both. The individual components are then directed upon an ion collector and discharged, and the intensity of the resultant ion current is measured. Thus, the several components of the sample may be caused to fall successively upon the collector by varying the electric or magnetic field, or by moving the collector successively into the respective paths of the several components.

In a method of mass spectrometry heretofore proposed, the ions produced by the bombardment of the gaseous sample by an electron beam have been formed into an ion beam and caused to flow in a semi-circular path through an evacuated envelope. The radius of this path was caused to vary with the ions of different mass-to-charge ratio by various means, and each ion beam of a given mass-to-charge ratio was successively directed to an ion collector located at the end of the semi-circular path.

The present invention involves a mass spectrometer which, although dependent on basic principles as hereinbefore described, involves the utilization of an entirely new type of control of the ion beams formed, and contemplates many advantages including small compactness in size, high resolution power, negligible variation in output due to irregularities of the collimator, or variations in density in the electron beam since all reach the collector.

The present invention contemplates a mass spectrometer comprising in combination an analyzing chamber, magnetic means for producing a magnetic field across the chamber means for causing ions formed by bombardment of the gas by an electron beam to travel generally along the lines of force of the magnetic field, and electrodes for producing an alternating field transversely to the flow of ions within said chamber, whereby certain of the ions which are in resonance with the frequency of the alternating field are caused to pursue an expanding spiral path while moving generally along the lines of force of the magnetic field, and a target electrode upon which those particular ions pursuing the expanding spiral path will impinge. The term "direction of the magnetic field" as used herein in the specification and claims is intended to indicate parallelism independent of polarity and direction of ion travel. Other ions, not in resonance with the frequency of the alternating field, are sent in a different path and do not impinge on the target.

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A feature of the invention is the provision of means for causing the spiral stream of ions to exist in pulsations. This may conveniently be done by varying the frequency of the alternating field at a predetermined rate. Preferably, the change of frequency should be sufficient to shift from the frequency of maximum ion reception for the particular ion mass unit being measured to the frequency of the minimum ion reception between that mass unit and the next adjacent mass unit.

The present invention may be more clearly understood with reference to the following detailed description taken in relation to the accompanying drawing, in which:

Fig. 1 is a horizontal cross-section of a mass spectrometer according to the invention, and showing the spiral path of ions of a mass-to-charge ratio which it is desired to collect;

Fig. 2 is an elevational cross-section of the mass spectrometer of Fig. 1 taken along the line 2—2 of Fig. 1;

Fig. 3 is a view similar to that of Fig. 2, but showing a path taken by other ions of a different mass-to-charge ratio than that illustrated in Figs. 1 and 2;

Fig. 4 is a graph illustrating the operation of the mass spectrometer.

Referring to Figs. 1 and 2 of the drawing, it will be observed that the mass spectrometer has an envelope 21. Certain auxiliary equipment, such as evacuating means, etc., are omitted as being conventional and unnecessary to an understanding of the present invention. A tube 11 is connected to the envelope and through it a sample of gas to be analyzed is admitted. The tube 11 terminates at the point of inlet in a chamber 12 provided with a hole 13 through which the molecules of the sample are admitted to the envelope 10.

An electron gun 14 is mounted within the envelope discharging a stream of electrons which pass through the envelope as an electron beam ionizing the molecules of the sample to be analyzed so they pass into the envelope counter-currently to the electron beam which passes

through the hole 13 of the chamber 12. However, it should be understood that the gas might be admitted from some other position or direction if desired.

An electron target 15 connected to ground is disposed within the chamber 12 to collect the electrons.

Disposed within the envelope 10 are the D. C. electrodes 16 and 17 provided with slits 18 and 19 in the path of the electron beam and of the slit 13. The electrode 16 is disposed adjacent to the chamber 12 with the slit 18 in juxtaposition to the slit 13 in the chamber. The two electrodes 16 and 17 are connected to a direct current power source 20.

To produce an alternating electric field across the space in the analyzer chamber formed by envelope 10, there are provided therein a plurality of A. C. electrodes 22, 23, 24, 26, 27, 28 and 29 of which 26 is seen in Fig. 1, and to produce a longitudinal magnetic field there are provided the magnetic poles 30 and 31.

The potential on the D. C. electrodes 16, 17 is sufficient to produce a steady electric field component in the direction from plate 16 to plate 17, so as to impart to the ions issuing from the hole 13 in the chamber 12 a component of velocity in the direction of the electrode 17. The effect of the transverse alternating field set up by the A. C. electrodes 23, 24, 26, 27 and 28 and the longitudinal magnetic field established by the magnetic poles 30 and 31 is to cause the ions of the particular mass-to-charge ratio which is in resonance with the alternating field, to pursue an expanding spiral path 40 in the direction of the electrode 17. In this regard, the particular ion component induced to travel in this spiral path will be dependent upon the frequency of the electric field and intensity of the magnetic field; and these fields cause those ions of corresponding resonance frequency to assume this spiral path longitudinally of the magnetic field, i. e. generally along its lines of force. The remaining ions having resonant frequencies not corresponding to the field frequency set up by the A. C. electrodes will travel toward electrode 17 in paths in the form of Lissajous figures extending transversely across the axis of travel toward electrode 17 and having an envelope small enough so as not to impinge on the collector electrode. This is represented by the path 41 in Fig. 3. These Lissajous figure paths are due to the combined effects of the alternating field and of the resonance of the ion itself which is a function of its specific mass and the intensity of the magnetic field.

The potential developed between electrode 16 and 17 produces as described above, a field across the chamber in the direction of the magnetic lines of force. It is this field which causes the ions to travel from the point of inlet toward the electrode 17 during which time they are acted upon by the magnetic and alternating fields so that the resonant ions follow a path in the form of an expanding spiral and the non-resonant ions travel toward electrode 17 in paths shaped in the form of Lissajous figures. It is desirable to cause these non-resonant ions to travel through the chamber, i. e. toward electrode 17 so as to prevent the accumulation of a space charge or ion "build-up" within the chamber which would interfere markedly with the operation of the device.

Thus in the spiral mass spectrometer herein set forth, the path of the ions of the resonant frequency γ_m is a spiral which enlarges its size without limit. The paths of all other ions are Lissajous figures centrally located within the analyzer chamber with reference to the central axis due to the interaction of the two frequencies corresponding to the applied voltage and the resonant frequency of the ion, respectively. In order for this mass spectrometer to operate at constant resolution, the size of the Lissajous figure envelope of the ions one mass unit different from those resonant ions induced to assume the spiral path should be maintained approximately constant and within the electrode structure in the analyzer chamber.

Interposed between the A. C. electrodes which establish the transverse alternating field there is located an ion collector 33 projecting into the path of the expanding spiral of ions of the given resonance frequency. In this fashion, the ions induced to form this spiral path may be separately counted, while those assuming the paths of a Lissajous figure will not impinge on the ion collector 33. By altering the frequency of the transverse alternating field, ions of correspondingly different resonance frequencies will be caused to assume the spiral path and may then be measured upon impingement on the ion collector.

In the operation of this spiral type mass spectrometer, it is desirable to frequency modulate the applied high frequency alternating voltage over a frequency range which causes the collector plate to receive ions first at maximum amplitude for a given mass unit and then for the minimum number of ions between that mass unit and the adjacent mass unit. This is accomplished by connecting the electrodes 22, 23, etc., to a frequency modulated oscillator 34, which "wobbles" or varies the frequency on the collector by the proper amount. As circuits for modulating or varying the frequency of an oscillator output at a regular rate are well known, no detailed description of the oscillator need be given here.

In the absence of frequency modulation of the oscillator, the oscillator output would be a constant frequency; and this could be adjusted to correspond with the resonance of a particular ion mass unit. For example, if an oscillator frequency f_1 corresponds with a particular ion of mass unit m_1, these ions of this particular mass unit will impinge on the collector and produce a corresponding collector current. This is represented in Fig. 4 which is a graph of oscillator frequency f against the collector current I. At frequency f_1 there will be produced the peak of current I_1 representing the collection of ions of mass m_1.

If the oscillator frequency were shifted somewhat to a different frequency f_2, corresponding to the resonance of an adjacent ion mass unit m_2, there will be produced a different peak of current I_2 corresponding to the number of ions of mass m_2 which are caused to form into the spiral path and impinge on the collector.

In between the frequencies f_1 and f_2 there is a frequency f_3 at which a minimum of ions is received at the collector. This minimum is not ordinarily zero, as there will ordinarily be some background level of reception of stray ions even in the absence of the particular mass units m_1 and m_2. This background level is represented by the dotted line b; and its level at frequency f_3 controls the collector current at f_2.

The frequency modulation applied to the oscillator output should preferably be such as to vary the oscillator frequency between f_1 and f_3 (or be-

tween f_1 and a high frequency of minimum current) at the frequency modulation rate. This will cause the ions of unit mass m_1 to shift back and forth from the spiral path to the Lissajous figure path at the modulation frequency rate. At the instants when the mass m_1 ions are in the spiral path at frequency f_1, maximum current is received, and at the instants when the frequency is at f_3, minimum current is received.

The reception of current at the collector **33** in pulsations in the manner just described is preferable in that such a current is more quickly and efficiently amplified than when the pulsations are not present.

Another possible method of operation would be to keep constant the size of the envelope of the path of an ion having a resonant frequency which is a given fraction of the applied frequency. As the equations below will show, this latter method of operation will be obtained if the magnitude of the high frequency voltage is made proportional to the square of the applied frequency.

$$\gamma_m=\frac{Be}{cm} \tag{1}$$

where:

γ_m=resonant frequency of mass m
ω=applied frequency
B=magnetic field strength, E. M. U.
$\frac{m}{e}$=mass-to-charge ratio, E. S. U.
c=velocity of light, cm./sec.

$$X=\frac{KE}{\gamma^2\left(1-\frac{\gamma}{\omega}\right)} \tag{2}$$

where

X=size of envelope of Lissajous figure, cm.
E=the electric field strength, E. S. U.

$$E=\frac{X_m\gamma_m^2}{K}\left(1-\frac{\gamma_m}{\omega}\right) \tag{3}$$

where the subscript m denotes a particular mass.

If, as stated above, it is desirable to hold the function γ_m/ω constant and X_m constant, E must vary proportionally to γ_m^2 which is in turn proportional to ω^2, since

$$\frac{\gamma_m}{\omega}$$

is constant.

As above stated, it is desirable to operate this spiral type mass spectrometer so as to produce a pulsating current at the ion collector which may be accomplished by the frequency modulation of the high frequency voltage as hereinbefore described. Equation 1 above shows that the ratio in magnitude of this variation in frequency to the applied frequency should be inversely proportional to the mass being recorded, in order that the pulsations shall involve alternately the receipt at the collector of the maximum number of ions of a given mass and the minimum number of ions between that mass and the next adjacent mass. For example, this ratio for a particular ion component of mass **58** should be in the order of magnitude of 1/116.

As an example of the use of the frequency modulation of the output of the oscillator, the oscillator frequency, in the absence of the modulation, might be established, for instance, at 1,000,000 cycles per second. To frequency modulate it most effectively for mass 58, the variation of this frequency should be about $\frac{1}{116}$ of 1,000,000 cycles, or about 8600 cycles. This could be done by varying the frequency between the limits of about 1,000,000 cycles and 991,400 cycles per second; or, what amounts to about the same thing, between 1,004,300 and 995,700 cycles per second. The rate of variation between the frequency limits may be set at some convenient rate such as 1,000 cycles per second.

Many modifications of the mass spectrometer of the present invention will occur to those skilled in the art as for example, in the type of electron gun to be employed, the method of amplification, the method of ionization, and the like without departing from the essential features of the invention which involve the utilization of forces adapted to induce the ions formed by whatever manner to travel in a spiral path when the resonant frequencies of such ions correspond to the frequency of the field established within the mass spectrometer and the measurement by a collector adapted to pick up only those ions of a given mass-to-charge ratio traveling in this spiral path, of the quantum of ions of any given mass-to-charge ratio. It is to be understood that any such modifications occurring to those skilled in the art are within the scope of the foregoing description and of the following claims.

We claim:

1. In a mass spectrometer the combination which comprises an analyzer chamber, means for producing an alternating electrical field across a space in the chamber, means for producing a magnetic field across the space transverse to the alternating electric field, and means for causing ions disposed in said space to travel generally along the lines of force of the magnetic field, whereby certain of such ions pursue an expanding spiral path in the space and a target electrode for ions disposed in the space in the expanding spiral path.

2. In a mass spectrometer the combination which comprises an analyzer chamber, means for producing an alternating electric field across a space in the chamber, means for producing a magnetic field across the space transverse to the alternating field, means for establishing a component of an electrical field along the lines of force of the magnetic field to cause ions disposed in said space to travel generally along the lines of force of the magnetic field, whereby certain of such ions pursue an expanding spiral path in the space and a target electrode for ions disposed in the space in the expanding spiral path.

3. In a mass spectrometer the combination which comprises an analyzer chamber, means comprising a plurality of electrode plates disposed within said analyzing chamber for producing an alternating electric field across a space in the chamber, means for producing a magnetic field across the space transverse to the alternating field, and means for causing ions disposed in said space to travel generally along the lines of force of the magnetic field, whereby certain of such ions pursue an expanding spiral path in the space and a target electrode for ions disposed in the space in the expanding spiral path.

4. In a mass spectrometer the combination which comprises an analyzer chamber, means for producing an alternating electric field across a space in the chamber, means for producing a magnetic field across the space transverse to the alternating field, means for admitting molecules to said space, an electron gun located in said chamber opposite said means for admitting molecules for firing electrons generally along the mag-

netic lines of force to ionize molecules as they are admitted to said space, and means for causing ions disposed in said space to travel generally along the lines of force of the magnetic field, whereby certain of such ions pursue an expanding spiral path in the space and a target electrode for ions disposed in the space in the expanding spiral path.

5. In a mass spectrometer the combination which comprises an analyzer chamber, means for producing an alternating electric field across a space in the chamber, means for producing a magnetic field across the space transverse to the alternating field, means for admitting molecules to said space, an electron gun firing an electron beam counter-current to the stream of entering molecules to ionize the molecules as they are admitted to said space, and means for causing ions disposed in said space to travel generally along the lines of force of the magnetic field, whereby certain of such ions pursue an expanding spiral path in the space and a target electrode for ions disposed in the space in the expanding spiral path.

6. In mass spectrometry involving the formation of ions and the separating thereof in a magnetic field, the improvement which comprises causing the ions to travel generally along the lines of force of the magnetic field while simultaneously subjecting them to the action of an alternating electrical potential applied transverse to the lines of force of the magnetic field, whereby the ions of a given mass-to-charge ratio tend to travel in expanding spiral paths so that those of different mass-to-charge ratios are separated from each other in space, and selectively collecting the ions thus separated.

7. In mass spectrometry involving the formation of ions and the separating thereof in a magnetic field, the improvement which comprises causing the ions to travel generally along the lines of force of the magnetic field responsive to a unidirectional electrical field generally along the lines of force of said magnetic field while simultaneously subjecting them to the action of an alternating electrical potential applied transverse to the lines of force of the magnetic field, whereby the ions of a given mass-to-charge ratio tend to travel in expanding spiral paths so that those of different mass-to-charge ratios are separated from each other in space, and selectively collecting the ions thus separated.

8. In mass spectrometry involving the formation of ions of a gas sample and the separating thereof in a magnetic field, the improvement which comprises forming said ions by subjecting said gas sample to a counter current electron beam, causing the ions to travel generally along the lines of force of the magnetic field while simultaneously subjecting them to the action of an alternating electrical potential applied transverse to the lines of force of the magnetic field, whereby the ions of a given mass-to-charge ratio tend to travel in expanding spiral paths so that those of different mass-to-charge ratios are separated from each other in space, and selectively collecting the ions thus separated.

9. In mass spectrometry involving the formation of ions of a gas sample and the separating thereof in a magnetic field, the improvement which comprises forming said ions by subjecting said gas sample to an electron beam, causing the ions to travel generally along the lines of force of the magnetic field responsive to a component of the electric field applied along the lines of force of said magnetic field and counter to said electron beam while simultaneously subjecting them to the action of an alternating electrical potential applied transverse to the lines of force of the magnetic field, whereby the ions of a given mas-to-charge ratio tend to travel in expanding spiral paths so that those of different mass to charge ratios are separated from each other in space, and selectively collecting the ions thus separated.

10. In mass spectrometry involving the formation of ions and the separating thereof in a magnetic field, the improvement which comprises causing the ions to travel generally along the lines of force of the magnetic field while simultaneously subjecting them to the action of an alternating and pulsating electrical potential applied transverse to the lines of force of the magnetic field, whereby the ions of a given mass-to-charge ratio tend to travel in expanding spiral paths of pulsating cross section so that those of different mass-to-charge ratios are separated from each other in space, and selectively collecting the ions thus separated.

11. In a mass spectrometer the combination which comprises an analyzer chamber, means for producing an alternating electric field of varying intensity across a space in said chamber, means for producing a magnetic field across the space transverse to said alternating field, and means for causing ions disposed in said space to travel generally along the lines of force of said magnetic field, whereby certain of said ions pursue an expanding spiral path the cross section of which varies in proportion to the variation in intensity of said alternating field and a target electrode for ions disposed in the space in the said expanding spiral path.

12. In a mass spectrometer, the combination which comprises an ionization chamber, an analyzer chamber, means for drawing ions from the ionization chamber along an axis through the analyzer chamber, electrostatic plates arranged around said axis and leaving a space at and around the axis for ion movement, means for applying an alternating voltage to said plates whereby ions in resonance with the frequency of said voltage travel in an expanding spiral path symmetrically around the axis, and a collector plate protruding closer to the axis than the other plates, whereby ions of said spiral path impinge on said collector plate, and ions other than those in resonance are confined within said space.

13. A combination according to claim 12 in which the electro-static plates are arranged symmetrically on either side of said axis, and the alternating voltage is applied across said plates on either side of the axis.

14. A combination according to claim 12 in which there are a plurality of said electro-static plates arranged at different distances from said axis, the plates further from the axis having greater alternating voltage than the plates nearer the axis.

15. In a mass spectrometer the combination which comprises an analyzer chamber, means for producing an alternating electric field across the space in the chamber, means for producing a magnetic field across the space transverse to the electric field, whereby ions introduced into said space travel along the path of the magnetic field, and the particular ions in resonance with the frequency of the alternating electric field pursue an expanding spiral path, and means for wobbling the frequency of said alternating field between

frequency limits, whereby the path of the resonant ions changes from the expanding spiral path to a Lissajous figure path within the space at the rate of the frequency wobble.

16. In a mass spectrometer, the combination which comprises a gas ionization chamber, an analyzer chamber, a magnetic field related to the analyzer chamber for drawing ions from the ionization chamber along an axis to the analyzer chamber, electrostatic plates arranged around said axis and leaving a space at and around the axis for ion movements, a frequency modulated oscillator connected to said plates, whereby the alternating field created by said plates is varied at the frequency modulated rate, a collector electrode within the analyzer chamber located closer to said axis than the electro-static plates, whereby said collector collects ions in resonance with the frequency during the times of the frequency modulation cycle when the oscillator frequency corresponds with the resonance frequency, and other ions not in resonance with said frequency are not collected by said collector.

17. In mass spectrometry involving the formation of ions and the separation thereof into different paths depending on their mass-to-charge ratio, the improvement which comprises drawing the ions through an analyzer chamber along a magnetic field and simultaneously subjecting them to the action of a frequency modulated alternating electrical field applied transverse to the direction of the magnetic field whereby the ions in resonance with the frequency of the electrical field travel in an expanding spiral while moving along the magnetic field, and pulsate between the spiral path and a Lissajous figure path in accordance with the frequency modulation rate, and collectively collecting the ions when they are traveling in the expanding spiral path.

18. A mass spectrometer comprising the combination of an envelope providing a closed space to which gases may be admitted, means for ionizing said admitted gases, means for generating a magnetic field which traverses said space, means including electrodes in said space for generating an alternating electric field oriented to be normal to said magnetic field and having a frequency of alternation corresponding to the natural frequency of ions possessing a certain mass whereby said ions may be accelerated in spiral paths, grid conductors within said space connected to maintain a desired alternating electric field distribution, electrode means disposed in said space in the path of ions of said certain mass, means for indicating the discharge upon said electrode means of ions which impinge thereupon, and means for removing from said space ions which possess natural frequencies other than that corresponding to the frequency of said electric field.

19. In mass spectrometry involving the formation of ions and the separation thereof in a magnetic field, the improvement which comprises simultaneously subjecting the ions to the magnetic field and to an alternating electrical field oriented to be normal to the magnetic field and having a frequency of alternation corresponding to the natural frequency of ions possessing a certain mass whereby said ions may be accelerated in spiral paths, selectively collecting said ions of certain mass, and separately removing from the field the ions which possess natural frequencies other than that corresponding to the frequency of the electric field.

20. In mass spectrometry involving the formation of ions of a gas sample and the separation thereof in a magnetic field, the improvement which comprises forming the ions by subjecting the gas sample to an electron beam, introducing the ions into the magnetic field and simultaneously subjecting them to an alternating electric field oriented to be normal to the magnetic field and having a frequency of alternation corresponding to the natural frequency of ions possessing a certain mass whereby the ions of said certain mass are accelerated in spiral paths, selectively collecting and discharging the ions of certain mass, measuring the current produced by such selective discharge of the ions of said certain mass, and selectively removing from the field ions which possess natural frequencies other than that corresponding to the frequency of the electric field.

21. In a mass spectrometer comprising the combination of an envelope providing a closed space to which gases may be admitted, means for generating a magnetic field which traverses said space, an electron gun located with relation to said space so as to fire electrons generally along the magnetic lines of force and countercurrently to the direction of gas flow into the space, means including electrodes in said space for generating an alternating electric field oriented to be normal to the lines of force of the magnetic field and having a frequency of alternation corresponding to the natural frequency of ions possessing a certain mass whereby the ions of said certain mass may be accelerated in spiral paths, grid conductors within the space connected to maintain a desired alternating electric field distribution, electrode means disposed in the path of ions of said certain mass, means for indicating the discharge upon said electrode means of ions which impinge thereupon, and means for removing from the space ions which possess natural frequencies other than that corresponding to the frequency of the electric field.

22. In a mass spectrometer, the combination which comprises an analyzer chamber, means for producing an alternating electric field across a space in the chamber, means for producing a magnetic field across the space transverse to the alternating field, means for admitting molecules to the space, an electron gun located in the chamber opposite the means for admitting molecules for firing an electron beam countercurrently to the stream of entering molecules to ionize the molecules as they are admitted to the space, means for establishing a component of the electrical field oriented generally along the lines of force of the magnetic field, which means causes ions disposed in the space to travel generally along the lines of force of the magnetic field, whereby certain of such ions pursue an expanding spiral path in the space, and a target electrode for ions disposed in the space in the expanding spiral path.

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The following references are of record in the file of this patent:

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Notice of Adverse Decision in Interference

In Interference No. 87,000 involving Patent No. 2,627,034, H. W. Washburn and C. E. Berry, Mass spectrometry, final judgment adverse to the patentees was rendered November 27, 1956, as to claims 18, 19, and 20.

[*Official Gazette May 21, 1957.*]

Disclaimer

2,627,034.—*Harold W. Washburn* and *Clifford E. Berry*, Pasadena, Calif. MASS SPECTROMETRY. Patent dated Jan. 27, 1953. Disclaimer filed May 15, 1957, by the assignee, *Consolidated Electrodynamics Corporation*; the inventors approving.

Hereby enters this disclaimer to claims 18, 19, and 20 of said patent.

[*Official Gazette June 11, 1957.*]