Oct. 7, 1952 G. D. PERKINS 2,613,323

MASS SPECTROMETRY

Filed Nov. 15, 1949

FIG. 1

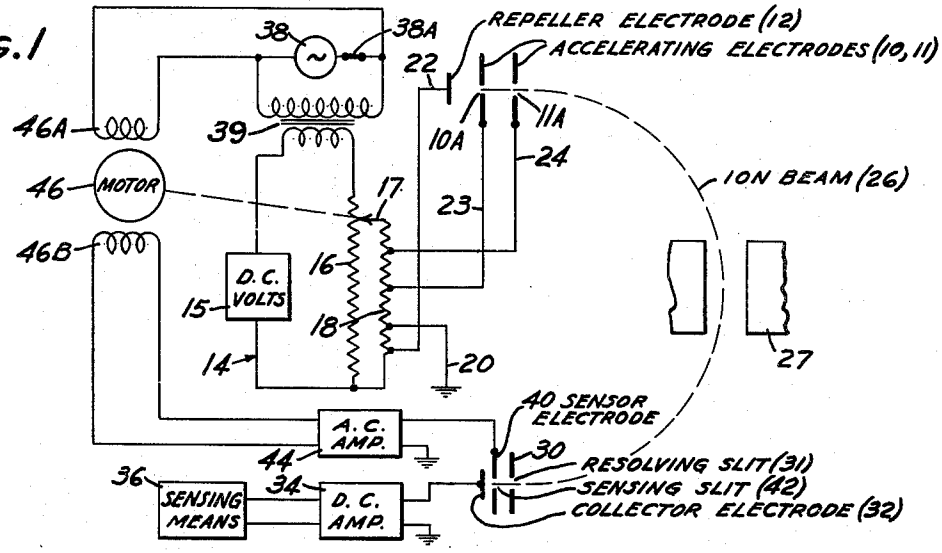


FIG. 2

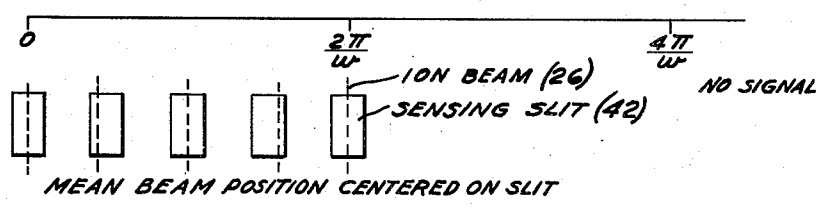


FIG. 2A

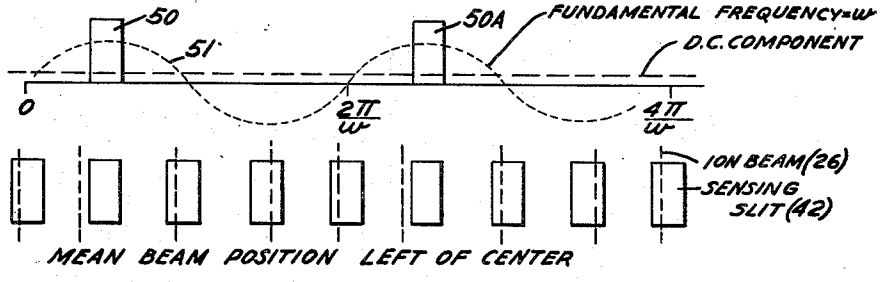
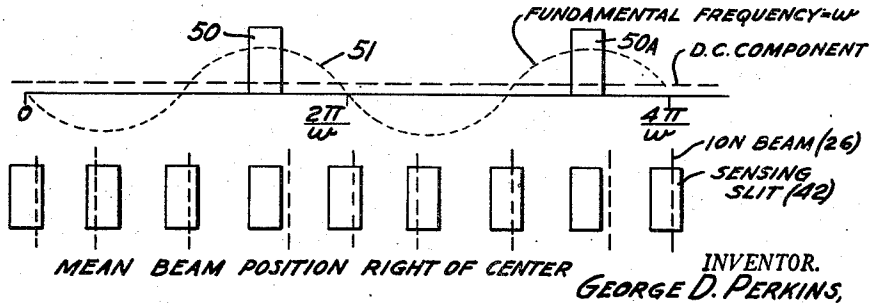


FIG. 2B



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Patented Oct. 7, 1952 2,613,323

UNITED STATES PATENT OFFICE

2,613,323

MASS SPECTROMETRY

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Application November 15, 1949, Serial No. 127,447

16 Claims. (Cl. 250—41.9)

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This invention relates to mass spectrometry and to mass spectrometers. More specifically, the invention is directed to improvements in the method of focussing ion beams and to apparatus for carrying out the improved method.

A mass spectrometer is an analytical instrument for sorting and measuring ions. Ordinarily, it includes an ionization chamber in which molecules of the sample to be analyzed are bombarded with a stream of electrons to convert them into ions. The ions formed are expelled from the ionization chamber by one or more repeller electrodes and are propelled by so-called propelling or accelerating electrodes, into and through an analyzer chamber. The analyzer chamber frequently takes the form of a curvilinear enclosure, say a tube. During passage through the chamber, the heterogeneous beam of ions is subjected to a transverse electrical or magnetic field, or both, to separate it into separate diverging ion beams, each of which is composed of ions of a given mass-to-charge ratio which differs from the mass-to-charge ratio of the ions forming the other beams. The diverging beams may be successively focussed through an exit or resolving slit in a focussing electrode onto a collector electrode. Alternatively, a single one of the ion beams may be continuously focussed on the resolving slit to the exclusion of the other beams. In either event, the focussing of the ion beam or beams is achieved by adjustment of the potential applied to the accelerating electrodes. The current produced by ion discharge at the collector electrode is indicative of the amount of ions in the particular beam and hence, constitutes a measure of the partial pressure of the molecules (from which the ions were derived) in the sample being analyzed.

As indicated above, it may be expedient to focus the mass spectrometer on a single mass peak or step-wise on two or more mass peaks rather than sweeping the diverging ion beams successively across the resolving slit. Such a "fixed focus" instrument is used for continuous monitoring where a continuous concentration of a single constituent in a sample stream is sought, in leak detection where the presence of an added contaminant is detected as it appears in the atmosphere surrounding a vessel under investigation, and generally under any circumstances in which a single component or a pre-selected group of components is to be measured.

For accurate analysis with a fixed focus mass spectrometer, it is important that the particular ion beam under investigation be at all times

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centered on the resolving slit. Any appreciable deflection of the subject beam from a centered position will affect the apparent intensity of the beam and hence, result in erroneous analysis. Since the factors which govern the position of the beam are the transverse magnetic field and the accelerating voltage, deviation of either of these variables from a pre-selected fixed value will result in displacement of the beam from a centered position.

It has been proposed to stabilize the accelerating voltage against deviations from a preselected value by means of a voltage-dropping network wherein a fraction of the accelerating voltage is compared with the voltage output of a standard cell. Any variation in proportionality between the fraction and the standard voltage is balanced automatically by a servo-motor or other means acting on the voltage source supplying the accelerating electrodes. However, the accuracy of such a system depends upon a resistance divider network, the long-term stability of which is questionable. Moreover, the possibility of loss of focus due to temperature sensitivity, variations in power supply to an electromagnet, or aging of a permanent magnet is in no way accounted for in such a voltage-dropping network.

I have developed a method for holding a given ion beam centered on the resolving slit which automatically compensates for fluctuations in the accelerating voltage and variations in the strength of the magnetic field. The invention contemplates in a method of mass spectrometry which comprises ionizing a sample to be analyzed, propelling the ions by means of a D. C. accelerating potential into an analyzer region, subjecting the ions in the analyzer region to a transverse magnetic field to separate them into individual ion beams, focussing one of said ion beams through a resolving slit in a terminal electrode, discharging the beam passing through the resolving slit, and sensing the current developed by discharge of the beam, the improvement comprising passing the ion beam through a sensing slit prior to discharging it, superimposing on the D. C. accelerating potential an A. C. component to cause the ion beam to oscillate transversely with respect the sensing slit, detecting uncentered oscillation of the beam and adjusting the accelerating potential to restore centered oscillation.

The invention also includes one form of apparatus for carrying out this method, which comprises in a mass spectrometer having an analyzer

chamber, ionization chamber, accelerating electrodes, a source of D. C. voltage connected to develop an accelerating potential on the accelerating electrodes, means for establishing a transverse magnetic field across the analyzer chamber, a collector electrode, and a terminal electrode having a resolving slit through which an ion beam is focussed on the collector electrode, the combination comprising a source of A. C. voltage connected to impress an A. C. component on the accelerating electrodes, a sensor electrode disposed intermediate the terminal electrode and the collector electrode and having a sensing slit therein, and means operative to vary the voltage applied from the D. C. source to the accelerating electrodes responsive to uncentered oscillation of the ion beam at the sensing slit to restore the beam to a centered position.

The invention will be more clearly understood by reference to the accompanying drawing in which:

Fig. 1 is a diagram of a mass spectrometer in accordance with the invention; and

Figs. 2, 2A, and 2B are charts showing, respectively, the results in terms of the output signal when the given ion beam is centered on the sensing slit, when the given ion beam is decentered to the left of the slit and when the ion beam is decentered to the right of the slit.

The mass spectrometer shown diagrammatically in Fig. 1 inculdes a pair of accelerating electrodes **10**, **11** and a repeller electrode **12**. Ions formed in the ionization region between the repeller electrode and the first accelerating electrode **10** are expelled from this region through a slit **10A** in the accelerating electrode by application of a positive potential to the repeller electrode. The expelled ions are accelerated between the electrodes **10** and **11** and are discharged from a slit **11A** in the second electrode by means of a potential impressed between the two accelerating electrodes.

The repelling and accelerating voltages are applied to the repeller and accelerating electrodes from a voltage supply circuit **14**, including a D. C. voltage source **15** connected in series with a slidewire resistor **16**. Resistor **16** is tapped by slider **17** which is connected through a second resistor **18** to the positive side of the D. C. source **15**. The resistor **18** is grounded at **20** adjacent the end thereof remote from the slider **17**. A lead **22** connects the repeller electrode to resistor **18** on the positive side of ground **20** and leads **23**, **24**, respectively, connect the two accelerating electrodes **10**, **11** to spaced points on the resistor **18** on the negative side of ground **20**. This arrangement represents a conventional voltage supply circuit for a fixed-focus mass spectrometer wherein any change in the voltage delivered from the source or applied across resistor **18** results in proportional change in the voltages applied to the two accelerating electrodes.

The ions issuing from slit **11A** in electrode **11** are caused to pursue a curved path as indicated by ion beam **26**, the curved path being produced as the result of a transverse magnetic field established by magnet **27**. A terminal electrode **30** is placed in the path of the ion beam, say after it has travelled through a 180° arc. A resolving slit **31** in the terminal electrode provides means whereby an ion beam of given mass-to-charge ratio may be focussed on a collector electrode **32** disposed behind the terminal electrode. Ions passing through the resolving slit **31** discharge on the collector electrode. The collector electrode is electrically connected to D. C. amplifier **34** which amplifies the discharge signal and feeds it to a sensing means **36** which may be a recorder or other sensing device. To this point the operation of the mass spectrometer of Fig. 1 is typical of so-called fixed-focus instruments.

In accordance with the present invention, a source **38** of A. C. voltage is coupled through a switch **38A** and a transformer **39** to the voltage supply circuit **14** to superimpose on the D. C. voltage applied to the accelerating electrodes a comparatively small A. C. component of a frequency ω.

A sensor electrode **40** is interposed between the focussing electrode **30** and collector electrode **32**. A sensing slit **42** is provided in the sensing electrode which is narrower than the resolving slit **31** but wider than the ion beam by an amount equal to a safe tolerance for departure of the ion beam from a centered position. Electrode **40** is connected to an A. C. amplifier **44**. A motor **46** is mechanically coupled to the D. C. voltage supply circuit **14**, as for example, to the slider **17** so that actuation of the motor controls the position of slider **17**, depending upon the direction of motor rotation. One coil **46A** of the motor is connected to the A. C. source **38** and the other coil **46B** of the motor is connected to the A. C. amplifier **44**.

In operation, the ion beam is oscillated by superimposing an A. C. voltage on the accelerating electrodes, the magnitude of the superimposed A. C. voltage being such as to develop beam oscillation having an amplitude less than the width of the sensing slit **42**. Under such circumstances, oscillation of the beam will have no effect on the D. C. component of the signal developed at the collector electrode. However, if either of the factors, namely, accelerating potential or magnetic field strength, varies sufficiently so that deviation of the beam from a centered position causes it to strike one side or the other of the sensing slit during oscillation, a signal will be produced in the sensing slit and introduced to the A. C. amplifier **44**.

The operation of the system of Fig. 1 is shown diagrammatically in Figs. 2, 2A and 2B under conditions wherein the ion beam is centered, displaced to the left of center, and displaced to the right of center, respectively, all with respect to the sensing slit **42**. In Fig. 2, the position of the ion beam **26** with respect to the sensing slit **42**, when the beam is oscillating about a centered position at frequency ω is shown. Hence, the zero position of the beam, as depicted at the extreme left of Fig. 2, is centered on the sensing slit and with an amplitude less than the width of the slit, the beam remains in the slit during oscillation and no signal is developed. In Fig. 2A, with the beam zero position to the left of center, a pulse is formed each time the beam falls outside the sensing slit. Such condition prevails in the quarter cycle immediately to the right of the zero position and again in the quarter cycle following the completion of a cycle, etc. Pulses **50**, **50A** are developed at the sensor electrode giving an A. C. curve **51** upon Fourier analyses, having a fundamental frequency ω. These pulses are amplified in the A. C. amplifier **44** and are fed to coil **46B** to drive motor **46** in the direction dependent upon the phase of the amplified pulses with relation to the A. C. signal developed at the source **38**. The pulses developed at the sensor electrode when the beam focus is to the right of center are shown in Fig. 2B to have the same fundamental wave

form and frequency but of exactly opposite phase from the pulses formed when the beam is off center to the left. When the pulse, shown in Fig. 2B, is amplified and applied across coil 46B, the motor 46 will be driven in direction opposite to that resulting from a pulse of the type shown in Fig. 2A.

In this manner, if the beam focus deviates from either side of center any appreciable amount, motor 46 will be immediately energized to adjust the position of slider 17 in a compensatory direction so that centered focus will be regained.

The method and apparatus described has the important advantage of correcting for all effects contributing to undesired loss of focus of the pre-selected ion beam. A high degree of sensitivity is not necessary in the A. C. amplifier since the device can be operated to center on a stronger peak than the particular one under investigation, if desired. Thus, if the particular peak of interest is weak, a stepper switch can be included in the D. C. supply circuit 14 to periodically alter the accelerating voltage to focus a stronger peak on the resolving slit. Under such circumstances, the A. C. voltage component can be applied to the accelerating electrode only when this stronger peak is in focus. Conveniently, this selectivity is obtained by ganging such a stepper switch to the switch 38A governing the application of an A. C. voltage from the source 38.

Constancy of amplification in the A. C. amplifier is also not a problem provided only that the gain remains large enough to drive the motor. The system is not adjusted responsive to the magnitude of the A. C. amplifier output so that variation in gain within the range sufficient to operate the motor can be tolerated. Furthermore, motors can be obtained commercially which automatically discriminate between noise and extraneous frequencies which, therefore, do not interfere with the operation of the apparatus so long as they are not present in sufficient amounts to jamb the motors by over damping.

The application of the invention is simpler, less expensive, and more certain than any means heretofore proposed for holding a mass spectrometer on the top of a pre-set peak or a group of peaks by attempting to achieve a high degree of long-term stability in the magnet and in the power supply.

Although the invention has been described with particular reference to a continuous monitor instrument in which a single mass peak is of interest, it is not so limited. In any operation wherein the mass peaks are focussed step-wise on the resolving slit, the present invention is of value.

Furthermore, application of the invention is not limited to the particular apparatus shown. It is entirely practical to produce the necessary oscillation of the ion beam by means other than impression of an A. C. voltage on the accelerating electrodes. For example, auxiliary electrodes arranged transversely with respect to the path of ion travel will induce the same type of oscillation when energized with an A. C. component. Alternatively, the magnetic field may be varied in a rhythmic manner for the same purpose. At the same time, means other than adjustment of the accelerating potential may be employed to maintain or restore centered focus independent of the particular expedient employed to oscillate the ion beam. Again, transversely arranged electrodes may serve this focussing function by impressing a D. C. potential thereon and varying this potential in the manner illustrated in conjunction with the accelerating electrodes. As still a third alternative, the magnetic field, whether developed by an electromagnet or permanent magnet, may be varied responsive to the signal developed by an off-centered oscillating beam.

The method of the invention, therefore, involves oscillating the ion beam in any desired manner and adjusting a beam focussing field whether it be a lateral or transverse electric field or a transverse magnetic field responsive to a characteristic signal developed by the oscillating beam when the mean position of the beam is not properly centered on the resolving slit.

I claim:

1. In mass spectrometry involving ionizing a sample to be analyzed, propelling the ions by means of a D. C. accelerating potential into an analyzer region, subjecting the ions in the analyzer region to a transverse magnetic field to separate them into individual ion beams, focussing one of said ion beams through a resolving slit in a terminal electrode, discharging the beam passing through the resolving slit and sensing the current developed by discharge of the beam, the improvement comprising passing the ion beam through a sensing slit after passage through the resolving slit and prior to discharging it, superimposing on the D. C. accelerating potential an A. C. component to cause the ion beam to oscillate transversely with respect to the sensing slit, detecting uncentered oscillation of the beam and adjusting the accelerating potential to restore centered oscillation.

2. In mass spectrometry involving ionizing a sample to be analyzed, propelling the ions by means of a D. C. accelerating potential into an analyzer region, subjecting the ions in the analyzer region to a transverse magnetic field to separate them into individual ion beams, focussing one of said ion beams through a resolving slit in a terminal electrode, discharging the beam passing through the resolving slit and sensing the current developed by discharge of the beam, the improvement comprising passing the ion beam through a sensing slit after passage through the resolving slit and prior to discharging it, superimposing on the D. C. accelerating potential an A. C. component to cause the ion beam to oscillate transversely with respect to the sensing slit and with an amplitude less than the width of the sensing slit, detecting uncentered oscillation of the beam and adjusting the accelerating potential to restore centered oscillation.

3. In mass spectrometry involving ionizing a sample to be analyzed, propelling the ions by means of a D. C. accelerating potential into an analyzer region, subjecting the ions in the analyzer region to a transverse magnetic field to separate them into individual ion beams, focussing one of said ion beams through a resolving slit in a terminal electrode, discharging the beam passing through the resolving slit and sensing the current developed by discharge of the beam, the improvement comprising passing the ion beam through a sensing slit after passage through the resolving slit and prior to discharging the beam, superimposing on the D. C. accelerating potential an A. C. component to cause the ion beam to oscillate transversely with respect to the sensing slit and with an amplitude less than the width of the sensing slit, detecting uncentered oscillation of the beam and adjusting

the accelerating potential responsive to the detected uncentered oscillation to restore centered oscillation.

4. In mass spectrometry involving ionizing a sample to be analyzed, propelling the ions by means of a D. C. accelerating potential into an analyzer region, subjecting the ions in the analyzer region to a transverse magnetic field to separate them into individual ion beams, focussing one of said ion beams through a resolving slit in a terminal electrode, discharging the beam passing through the resolving slit and sensing the current developed by discharge of the beam, the improvement comprising passing the ion beam through a sensing slit in a sensing electrode between the terminal and collector electrodes, periodically superimposing on the D. C. accelerating potential an A. C. component to cause the ion beam to oscillate transversely with respect to the sensing slit and with an amplitude less than the width of the slit whereby the oscillating beam generates an A. C. signal at the sensing electrode when it is not centered on the sensing slit, and adjusting the accelerating potential responsive to said A. C. signal to restore centered oscillation.

5. In mass spectrometry involving ionizing a sample to be analyzed, propelling the ions by means of a D. C. accelerating potential into an analyzer region, subjecting the ions in the analyzer region to a transverse magnetic field to separate them into individual ion beams, focussing one of said ion beams through a resolving slit in a terminal electrode, discharging the beam passing through the resolving slit and sensing the current developed by discharge of the beam, the improvement comprising passing the ion beam through a sensing slit in a sensing electrode between the terminal and collector electrodes, continuously superimposing on the D. C. accelerating potential an A. C. component to cause the ion beam to oscillate transversely with respect to the sensing slit and with an amplitude less than the width of the slit whereby the oscillating beam generates an A. C. signal at the sensing electrode when it is not centered on the sensing slit, and adjusting the accelerating potential responsive to said A. C. signal to restore centered oscillation.

6. In a mass spectrometer having an analyzer chamber, ionization chamber, accelerating electrodes, a source of D. C. voltage connected to develop an accelerating potential on the accelerating electrodes, means for establishing a transverse magnetic field across the analyzer chamber, a collector electrode, and a terminal electrode having a resolving slit through which an ion beam is focussed on the collector electrode, the combination comprising a source of A. C. voltage connected to impress an A. C. component on the accelerating electrodes to cause oscillation of the ion beam, a sensor electrode disposed intermediate the terminal electrode and the collector electrode and having a sensing slit therein and means operative to vary the voltage applied from the D. C. source to the accelerating electrodes responsive to uncentered oscillation of the ion beam at the sensing slit to restore the beam to a centered position.

7. In a mass spectrometer having an analyzer chamber, ionization chamber, accelerating electrodes, a source of D. C. voltage connected to develop an accelerating potential on the accelerating electrodes, means for establishing a transverse magnetic field across the analyzer chamber, a collector electrode, and a terminal electrode having a resolving slit through which an ion beam is focussed on the collector electrode, the combination comprising a source of A. C. voltage connected to impress an A. C. component on the accelerating electrodes to cause oscillation of the ion beam, a sensor electrode disposed intermediate the terminal electrode and the collector electrode and having a sensing slit therein, means for developing an A. C. signal responsive to uncentered oscillation of the beam with respect the sensing slit, and means operative to vary the voltage applied from the D. C. source to the accelerating electrodes responsive to said A. C. signal to restore the beam to a centered position.

8. In a mass spectrometer having an analyzer chamber, ionization chamber, accelerating electrodes, a source of D. C. voltage connected to develop an accelerating potential on the accelerating electrodes, means for establishing a transverse magnetic field across the analyzer chamber, a collector electrode, and a terminal electrode having a resolving slit through which an ion beam is focussed on the collector electrode, the combination comprising a sensor electrode disposed intermediate the terminal electrode and the collector electrode and having a sensing slit therein a source of A. C. voltage connected to impress an A. C. component on the accelerating electrodes to cause oscillation of the ion beam, the source of A. C. voltage being adapted to produce an A. C. component at the accelerating electrodes of such magnitude as to cause beam oscillation of an amplitude less than the width of the sensing slit, means for developing an A. C. signal responsive to uncentered oscillation of the beam at the sensing slit, and means operative to vary the voltage applied from the D. C. source to the accelerating electrodes responsive to said A. C. signal to restore the beam to a centered position.

9. In a mass spectrometer having an analyzer chamber, ionization chamber, accelerating electrodes, a source of D. C. voltage connected to develop an accelerating potential on the accelerating electrodes, means for establishing a transverse magnetic field across the analyzer chamber, a collector electrode, and a terminal electrode having a resolving slit through which an ion beam is focussed on the collector electrode, the combination comprising a source of A. C. voltage connected to impress an A. C. component on the accelerating electrodes to cause oscillation of the ion beam with respect to the resolving slit, a sensor electrode disposed intermediate the terminal electrode and the collector electrode and having a sensing slit therein, an A. C. amplifier connected to the sensor electrode and means operative to vary the voltage applied from the D. C. source to the accelerating electrodes responsive to the output of said A. C. amplifier.

10. In mass spectrometry involving ionizing a sample to be analyzed, propelling the ions by means of a D. C. accelerating potential into an analyzer region, subjecting the ions in the analyzer region to a transverse magnetic field to separate them into individual ion beams, focussing one of said ion beams through a resolving slit and sensing the current developed by discharge of the beam, the improvement comprising passing the ion beam through a sensing slit after passage through the resolving slit and prior to discharging it, superimposing on the D. C. accelerating potential an A. C. component to cause

the ion beam to oscillate transversely with respect to the sensing slit, detecting uncentered oscillation of the beam and adjusting the accelerating potential to restore centered oscillation.

11. In mass spectrometry involving ionizing a sample to be analyzed, propelling the ions by means of a D. C. accelerating potential into an analyzer region, subjecting the ions in the analyzer region to a transverse magnetic field to separate them into individual ion beams, focussing one of said ion beams through a resolving slit in a terminal electrode, discharging the beam passing through the resolving slit and sensing the current developed by discharge of the beam, the improvement comprising passing the ion beam through a sensing slit after passage through the resolving slit and prior to discharging it, superimposing on the D. C. accelerating potential an A. C. component to cause the ion beam to oscillate transversely with respect to the sensing slit and with an amplitude less than the width of the sensing slit, detecting uncentered oscillation of the beam and adjusting the accelerating potential to restore centered oscillation.

12. In mass spectrometry involving ionizing a sample to be analyzed, propelling the ions by means of a D. C. accelerating potential into an analyzer region, subjecting the ions in the analyzer region to a transverse magnetic field to separate them into individual ion beams, focussing one of said ion beams through a resolving slit in a terminal electrode, discharging the beam passing through the resolving slit and sensing the current developed by discharge of the beam, the improvement comprising passing the ion beam through a sensing slit after passage through the resolving slit and prior to discharging the beam, superimposing on the D. C. accelerating potential an A. C. component to cause the ion beam to oscillate transversely with respect to the sensing slit and with an amplitude less than the width of the sensing slit, detecting uncentered oscillation of the beam and adjusting the accelerating potential responsive to the detected uncentered oscillation to restore centered oscillation.

13. In mass spectrometry involving ionizing a sample to be analyzed, propelling the ions by means of a D. C. accelerating potential into an analyzer region, subjecting the ions in the analyzer region to a transverse magnetic field to separate them into individual ion beams, focussing one of said ion beams through a resolving slit in a terminal electrode, discharging the beam passing through the resolving slit and sensing the current developed by discharge of the beam, the improvement comprising passing the ion beam through a sensing slit in a sensing electrode located between the terminal and collector electrodes, periodically superimposing on the D. C. accelerating potential an A. C. component to cause the ion beam to oscillate transversely with respect to the sensing slit and with an amplitude less than the width of the slit whereby the oscillating beam generates an A. C. signal at the sensing electrode when it is not centered on the sensing slit, and adjusting the accelerating potential responsive to said A. C. signal to restore centered oscillation.

14. In mass spectrometry involving ionizing a sample to be analyzed, propelling the ions by means of a D. C. accelerating potential into an analyzer region, subjecting the ions in the analyzer region to a transverse magnetic field to separate them into individual ion beams, focussing one of said ion beams through a resolving slit in a terminal electrode, discharging the beam passing through the resolving slit and sensing the current developed by discharge of the beam, the improvement comprising passing the ion beam through a sensing slit in a sensing electrode located between the terminal and collector electrodes, continuously superimposing on the D. C. accelerating potential an A. C. component to cause the ion beam to oscillate transversely with respect to the sensing slit and with an amplitude less than the width of the slit whereby the oscillating beam generates an A. C. signal at the sensing electrode when it is not centered on the sensing slit, and adjusting the accelerating potential responsive to said A. C. signal to restore centered oscillation.

15. In mass spectrometry involving ionizing a sample to be analyzed, propelling the ions by means of a D. C. accelerating potential into an analyzer region, subjecting the ions in the analyzer region to a transverse magnetic field to separate them into individual ion beams, focussing one of said beams through a resolving slit, discharging the beam passing through the slit and sensing the current developed by discharge of the beam, the improvement comprising passing said ion beam through a sensing slit after it has passed through the resolving slit, causing the beam to oscillate transversely with respect to the sensing slit, detecting uncentered oscillation of the beam and altering the beam focus to restore centered oscillation.

16. In a mass spectrometer having an analyzer chamber, ionization chamber, accelerating electrodes for propelling ions from the ionization chamber through the analyzer chamber, means for establishing a transverse magnetic field across the analyzer chamber, and a collector electrode separated from the analyzer chamber by an auxiliary electrode having a resolving slit therein, the combination comprising a sensor electrode having a sensing slit therein and disposed between the auxiliary and collector electrodes, means for oscillating an ion beam with respect to said sensing slit, and means operable responsive to uncentered oscillation of the beam to vary the beam focus to restore centered oscillation of the beam.

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No references cited.