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2,643,341

MASS SPECTROMETER ION SOURCE

Filed March 3, 1948

2 Sheets-Sheet 1

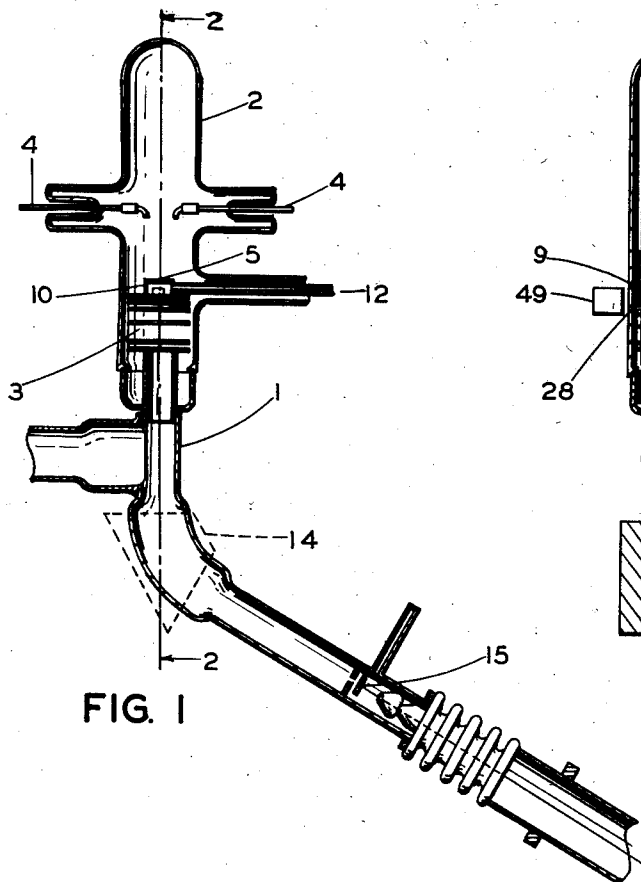


FIG. 1

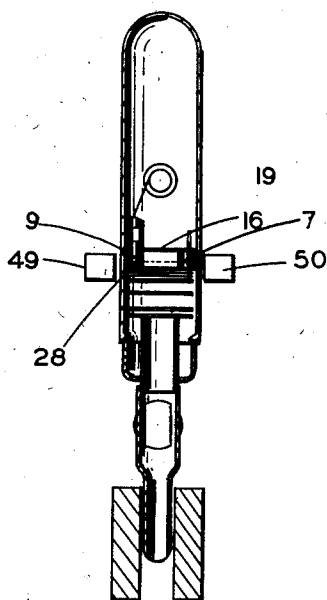


FIG. 2

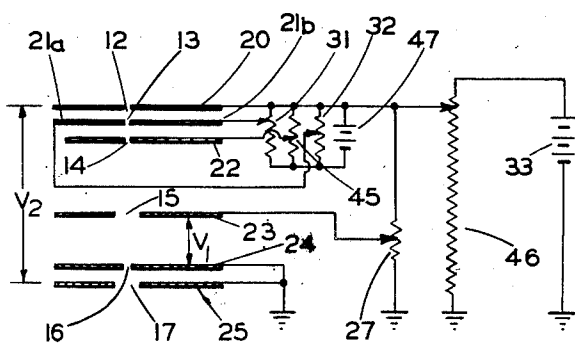


FIG. 5

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2 Sheets-Sheet 2

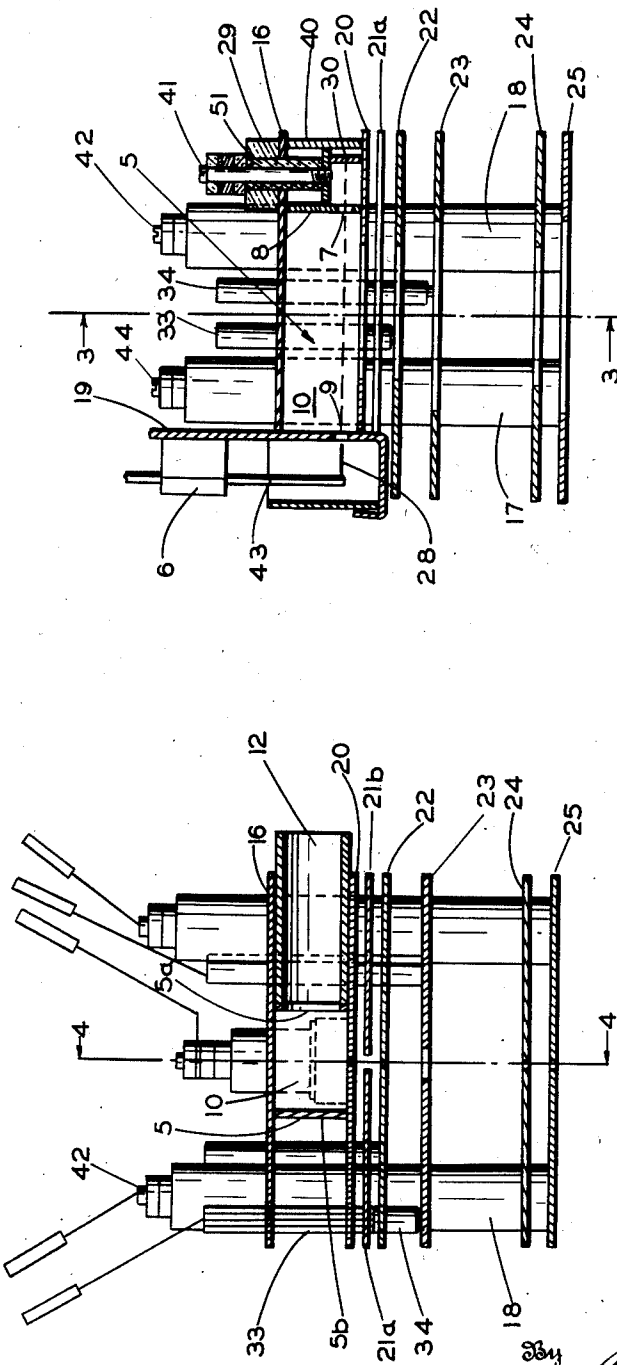


FIG. 4

FIG. 3

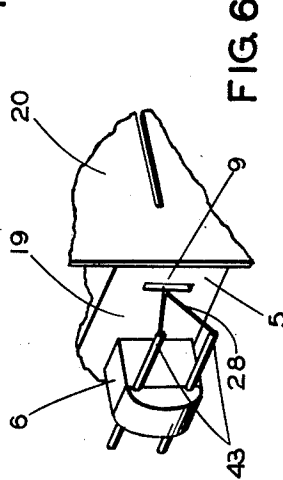


FIG. 6

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MASS SPECTROMETER ION SOURCE

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mesne assignments, to the United States of
America as represented by the United States
Atomic Energy Commission

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5 Claims. (Cl. 250—41.9)

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The present invention relates generally to thermionic ion sources, and more particularly to an improved method and apparatus for ejecting the ions from the ion chamber of a mass spectrometer and for selectively focusing ions of different masses.

It is well known that a gas may be bombarded by a stream of electrons, and that a certain number of its molecules become ionized as a consequence thereof. If a gaseous mixture is similarly treated, a mixture of ions of various kinds will result. Such a mixture of ions may be subjected to the action of a combination of electrostatic and magnetic fields to cause those ions having a particular mass to impinge upon a collecting member to which electrons will flow, thus creating an electric current. This current may be amplified and used as a measure of the number of ions having the aforementioned particular mass.

A mass spectrometer is a device adapted to separate components of a neutral gaseous mixture fed into it by bombarding such mixture with a stream of electrons, accelerating the resulting ions by passing them through a drop in electrical potential to focus and direct them into a magnetic field where ions having the same mass are bent to follow the same path. Thus, due to the influence of the magnetic field, the ions divide themselves into groups, each group containing only ions of the same mass and each group following a different path. Ions of any particular path may be projected upon and made to strike a collector, where the current flowing from the plate thereof serves as a measure of the quantity of such ions. Two factors determine the path of the ion in focusing upon the collector plate, the mass of the ion and its acceleration. Thus the mass spectrometer may be adjusted by changing the accelerating potential to collect ions having a given mass; and the number of the ions having this mass is proportional to the molecules from which the ions were produced. Accordingly, it is entirely feasible to employ the mass spectrometer as a gas analyzing means.

It can be shown mathematically that in order to focus a stream of ions through aligned slits in a series of spaced plates, the intermediate plates should be held at a potential which is some definite proportion of the potential drop across the entire series of plates. However, it is desirable to gather as many ions as possible into the focusing slits. Heretofore, efforts were made to accomplish both of these results by placing a plate outside of the focusing device and holding it at a potential intended to drive the ions into the

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initial slit while maintaining all of the intermediate plates at carefully calculated proportions of the total voltage drop.

One disadvantage of former devices was that they operated poorly at low potential differences across the focusing mechanism. The focusing devices were ineffective at such low potentials so that ions were lost because of improper focusing. Ions were also lost because they were not gathered into the focusing mechanism, in addition to the normal loss of some ions because of such things as stray currents in the device. All of this led to grave difficulties in attempting to utilize spectrometers with conventional ion sources therein for a wide range of potentials across the focusing mechanism. If one plotted the current at the ion collecting plate against the voltage applied across the focusing means, varying the magnetic field to record the same ion beam over this range of voltages, former devices might develop a certain ion current "a" at 100 volts. They might also develop a current value of "50a" or more at 1000 volts. It should be remembered that the number of ions in the ionization chamber or its equivalent remains substantially the same where the electron emission at the filament is held constant. Therefore, former devices showed greatly varying efficiency or sensitivity due to defects in or irregular operation of the focusing mechanism. Former spectrometers could not be efficiently used over a wide range and were therefore inadequate for continuous analysis and as a recording means.

It is, therefore, a principal object of the present invention to provide an ion source which may be adjusted over a wide range of potential differences, and therefore one which may be used over a wide range of masses without loss of sensitivity and without seriously changing the intensity of the ion current because of this adjustment.

It is another object of the invention to provide an ion source in which more effective focusing of the ions is achieved thereby reducing to a minimum the loss of ion so as to provide a relatively intense ion current permitting more effective measurement of the ions.

Another object of the invention is to provide an ion source having an ionizing chamber of an improved construction, permitting the use of a higher gas pressure, and which, when used in connection with an improved filament structure also provides more effective production of ions without excessively increasing the ionizing potential.

It is a still further object of this invention to

provide an ion source which may be effectively used over a wide range of focusing potentials without materially altering the ion current and may therefore be effectively employed for analyzing many different mixtures containing elements having a wide range of masses.

Other objects and advantages of my invention will appear from the following specification and accompanying drawings, and the novel features thereof will be particularly pointed out in the annexed claims.

Fig. 1 is a schematic or diagrammatic view indicating one form of mass spectrometer with which the present ion source may be employed showing in vertical section one embodiment of the invention installed therein.

Fig. 2 is a fragmental sectional view of the spectrometer incorporating my improved ion source, taken along the line 2—2 of Fig. 1, but in a plane displaced angularly 90° from the showing of that figure.

Fig. 3 is a longitudinal sectional elevation of the source used in my improved mass spectrometer.

Fig. 4 is a longitudinal sectional elevation of the same source taken along the line 4—4 of Fig. 3, in a plane displaced 90° from that of Fig. 3.

Fig. 5 is a diagrammatic showing of the plates and the circuits connecting them in the improved ion source of my spectrometer.

Fig. 6 is a fragmental detail of the ion source showing my improved filament.

As pointed out hereinbefore, an ion source embodying the present invention may be used in connection with a device or process in which it is required to produce a stream of ions from a gaseous substance. However, my ion source has been used chiefly in connection with a mass spectrometer. Accordingly, since this application of my ion source is the preferred embodiment of my invention, it will be described herein chiefly when thus used.

Referring particularly to Fig. 1 of the drawings, disclosing the conventional spectrometer structure inclosed in an evacuated tube, 1 designates the main body of the tube through which the ion beams travel, and which is preferably made of copper tubing. To one end of the tube 1 is joined a glass envelope 2. Disposed within the envelope 2 and mounted on tube 1 is an ion source 3. The various electrical leads 4, 4 which pass through the envelope 2 are shown, but their connections to the different source elements have been omitted. This is done for purposes of clarity and because such connections are conventional.

Source 3 includes an elongated inclosure generally designated 5 which extends transversely of the envelope 2 and defines a chamber 10. The inclosure 5 shown in detail in Fig. 3 is defined by upper plate 16, lower plate 20, one pair of opposite elongated spaced walls 5a, 5b, and another pair of opposite walls 19, 40. In communication with an opening in one vertical side wall 5a is a tube 12 which passes out through the envelope 2 and serves as a sample tube for supplying neutral gases to the ionizing chamber 10 for analysis. Mounted outside of chamber 10 on leads 43, 43 is a V-shaped filament 28 shown in detail in Fig. 6. The V-shaped filament bridges the lower extremities of the leads 43, 43 which are supported on bracket 19, an extension of one wall of the inclosure 5, by insulator elements 6 (see also Figs. 2 and 4). Formed in the lower extremity of the bracket 19 in alignment with the filament 28 and slit 7 in the opposite partition wall 8 of inclosure

5 is a slot 9. Disposed within the chamber defined by the partition 8 and the inclosure walls including end wall 40 is a trap or anode 30 supported by stud 41 from the upper plate 16 through insulator 29 and insulating sleeve 51. The trap is maintained at a positive potential of 75 to 100 volts above the filament. Positioned beneath the lower wall or plate 20 of the ionizing chamber 10 are a series of electrically isolated spaced plates 21a, 21b, 22, 23, 24, and 25. These plates are held in spaced relation by means of glass spacers, two of which are designated as 17 and 18, carried by bolts 42, 44 which thread the various spacers and extend through appropriately dimensioned openings in the plates so as to be spaced and insulated therefrom. Nuts may be applied to the bolts 42, 44, and the whole structure may be held in assembled relation in a manner common to the conventional spectrometer. It is, of course, possible for structural details to vary, but some system of positioning these plates, as shown, for electrically isolating each plate and for aligning the slots must be employed. Positioned externally of the envelope 2 on either side of the source in line with the electron flow are the poles 49, 50 of a magnet for setting up a magnetic field along the ionizing chamber. In Figs. 3 and 4 insulating tubes such as 33, 34 which serve to house the leads running to plates 22 and 23 of the ion source 3 are generally employed. Other plates are fed in a similar manner.

The construction of the filament in the present device, which is in the form of a long narrow V coming to a point, is also advantageous in that it tends to concentrate the emission of electrons at the point. This feature, together with the presence of a magnetic field across the chamber 10, causes electrons to flow in a well-defined beam through the chamber 10 to the receiving means or trap 30. As a result the electron beam of the present device is much more effective.

For a relatively small gas inflow and low electron emission, the present device will produce more ions and therefore more ion current for measurement. This increases the accuracy of the spectrometer and also aids in increasing its resolution, for any focusing device is more effective with more ions flowing through it.

The structure of the present device, especially the chamber 10, permits the use of somewhat higher gas pressures in the spectrometer than in former spectrometers. This in turn provides a higher gas sample to gas background ratio. This is advantageous since some background gas is necessarily present although it is undesirable.

Within the chamber 10, the gaseous substance is bombarded with a stream of electrons passing from the filament to the anode or trap and a certain percentage of its molecules becomes ionized as a consequence. These ions pass or are withdrawn from the ionizing chamber and are directed into the aligned openings in the focusing and accelerating plates of the ion source in the manner more fully discussed hereinafter.

As a result of these operations, the ions are formed into a stream or beam and are directed linearly down the casing 1 toward the magnetic field indicated at 14. The magnetic field 14 extending perpendicular to the plane of Fig. 1 is preferably set up by one or more permanent magnets so constructed as to provide the generally trapezoidally shaped field indicated in Fig. 1. The ions are influenced by the magnetic field so that their paths are curved, the radius of curvature depending upon their mass and velocity.

Accordingly, it is possible to adjust the mass spectrometer to direct ions of a certain mass to a suitable collecting means such as the plate shown at 15.

In order that the present ion source may operate effectively, it is necessary to provide differences in electrical potential between various plates of the series of plates mentioned. A preferred wiring circuit and voltage applying means for carrying out this purpose is shown in Fig. 5. As there indicated, the voltage differences between the plates 20 and 25 is supplied by a source of microcurrent indicated at 33, with plate 20 being maintained at a positive potential with respect to plate 25. Since this difference in potential may be adjusted over a wide range, such for instance as from 100 volts to 2500 volts, it is preferable that this source 33 be of a type especially designed to provide high voltage. This may be done by conventional voltage amplifying means together with suitable variable resistances or a series of fixed resistances which may be placed in or out of the circuit as may be found necessary.

Plates 21a, 21b form a "split pair," i. e., they are half discs placed side by side but spaced so as to provide the slit 13 which coincides with slit 12 in the lower plate or shield 20 of the ionizing chamber of the source 3 of Fig. 1. Plates 22 and 23 are whole discs possessing slits or openings 14, 15. Plates 24, 25 are connected together and have aligned slits 16, 17. One lower plate 25 would suffice but two plates 24, 25 are provided to strengthen the structure. A difference in potential between plates 20 and 21b of about 100 volts is applied through the circuit of Fig. 5 so that the plate 20 is at a positive potential with respect to plate 21b. This same potential is maintained between plates 20 and 22. The potential difference between plates 20 and 21a is substantially the same. These potential differences are maintained substantially constant despite the wide range of adjustment of the potential drop between plates 20 and 24, 25, and such voltage control is brought about by the arrangement of resistances shown in Fig. 5. The potential of plates 21b and 21a may be adjusted by the variable resistances 31, 32 which bridge across voltage source 47 although it is possible to hold one of these resistances constant and vary the other one. By either of these means the potential may be balanced on the split plates 21a, 21b so that the same potential may be placed on each plate despite unavoidable defects or structural imperfections. If desired, however, the plates 21a and 21b may have somewhat different potentials, say about five volts, to overcome any slight tendency of the ions to stray or the beams to bend due to the magnetic field set up between the poles 43, 49 of the magnet. In addition, a tandem switch may be provided for adjusting the potential on plates 21a, 21b simultaneously, thereby making the potential of the unit consisting of plates 21a, 21b adjustable without effecting the relative potential between them. The potential of plate 22 is controlled by the resistance 45. As indicated, the voltage for plates 21a, 21b, and 22 is supplied by a voltage source 47, different from that for the rest of the plates. While this source is only indicated schematically, it may preferably comprise a suitable power or rectifying unit with appropriate voltage regulator tubes. By this means, the voltage between plates 20 and 21a, 20 and 21b, and 20 and 22 may be held constant while the voltage between plates 20 and 24, 25, is

varied over a wide range. Adjustment of the potential of the plate 23 is accomplished by adjustment of the moving contact on voltage divider 27. This resistance or its equivalent forms a part of the high voltage supply circuit mentioned previously in connection with the voltage source for the plate 20. Once this resistance 27 is adjusted to provide a proportional voltage gradient between plates 20 and 24, 25, the potential of plate 23 is maintained at the desired proportion of the voltage drop between plates 20 and 24, 25 even though the latter voltage drop may be varied widely by regulating the potentiometer or voltage divider 46.

The above arrangement holds the plates 21a, 21b and 22 at a constant potential with respect to plate 20. This feature is believed to be a radical departure from known practices and establishes a constant field which reaches through the slit 12 into the chamber 10 and attracts the ions formed therein into the focusing device of plates 20, 23, 24, and 25. This method has proven to be much more effective than former methods for gathering ions into the slits. Of course, this constant potential difference to some extent detracts from the focusing ability of the device. But it will be noted that the plate 23 is maintained at a potential proportional to the potential drop from 20 to 25. Satisfactory focusing may be obtained if the potential differences between the plates 20 and 21a, 20 and 21b, and 20 and 22 are maintained at a low value as compared with the voltage between plates 20 and 25. Accordingly, the present device is more effective when the voltage between plates 20 and 25 is high, but will operate satisfactorily when this voltage is as low as 100 volts. In order to produce as strong a field as possible without unduly raising the voltages at 21a, 21b and 22, these plates are placed close together and close to the shield 20.

While difficulty has been encountered in prior structures in securing proper focusing at low potentials, this is largely overcome in my arrangement. For example, the present device will produce an ion current "a" at 100 volts across the focusing mechanism and approximately "2a" at 1000 volts instead of "50a," which often resulted in the arrangements of the prior art. Accordingly, it is seen that this device has relatively constant operation over a wide range of potentials between plates 20 and 24, 25; therefore, it may be used over a wide range of masses and consequently for analyzing a large number of substances.

Having thus described my invention, I claim:

1. Apparatus for producing and focusing ions which comprises an ionization chamber, means for feeding vapors of a substance thereto, a filament, means for receiving electrons, said filament and said means being positioned near opposite sides of said chamber thereby causing electrons to pass thereacross for forming ions from said substance, a plurality of electrically isolated plates spaced from each other exteriorly of said chamber, a slot in a wall of said chamber, openings through said plates aligned with each other and with said slot, and voltage regulating means electrically connected with said wall and with said plates for establishing a fixed difference in potential between said wall and at least one plate adjacent thereto and also establishing differences in potential between said wall and others of said plates, said voltage regulating means being controllable for adjusting the potentials of the others of said plates to desired constant values while

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maintaining the potential of said one plate substantially fixed, whereby to provide a uniform potential gradient along said other plates.

2. A system of the character described comprising walls defining an ionization chamber, means for feeding a gaseous mixture to the chamber, a source for supplying electrons to ionize said mixture, an opening in the chamber, electrodes positioned adjacent the mouth of the opening and at progressively greater distances from the chamber, said electrodes being maintained at a low fixed potential with respect to said walls for removing ions from said chamber, a series of spaced plates positioned adjacent said electrodes for defining a passage way aligned with said opening, and means for applying progressively larger potential differences between each of the plates of said series, whereby to set up a substantially uniform potential gradient thereacross and provide high sensitivity over a wide range.

3. A system of the character described comprising an ionization chamber having an exit opening, means for feeding neutral gaseous vapors to said chamber, a source of electrons disposed within the chamber for providing electrons to ionize said vapors, a pair of electrodes positioned at the mouth of said opening to provide a passage way for the ions, a disk positioned beyond the spaced electrodes having a slot aligned with the passage way, means for impressing small fixed potentials of substantially the same magnitude on said electrodes and said disk for setting up a substantially constant electrostatic field to eject said ions, a series of spaced plates positioned beyond the disk at progressively greater distances from said chamber for defining a collimating channel, and means for impressing potentials on the plates corresponding in magnitude to their distances from the chamber for setting up a substantially uniform potential gradient to focus and accelerate the ions into a beam.

4. An ion source of the character described comprising walls defining an ionization chamber, means for feeding a gaseous mixture to the chamber, a source for supplying electrons to said chamber for bombarding the gaseous mixture and producing ions, an exit opening in the walls of said chamber for the passage of ions, a group of spaced slotted plates positioned externally of the chamber and adjacent the exit opening at progressively greater distances therefrom for defining a passage way for the ions, said plates being maintained at a small fixed potential with respect to

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the walls of said chamber for removing the ions through the exit opening, a second group of plates each spaced progressively further from the chamber and having slots therein aligned with said passage way, said second group of plates being maintained at potential differences with respect to the walls of said chamber corresponding in magnitude to their distances from the chamber, whereby to provide a substantially uniform potential gradient thereacross for focusing and accelerating the ions into a beam.

5. An ion source of the character described comprising walls defining an ionization chamber, means for feeding a gaseous mixture to the chamber, a source for supplying electrons to the chamber for bombarding the gaseous mixture to produce ions, an exit opening in the walls of said chamber for the passage of ions, a group of spaced slotted electrodes positioned externally of the chamber at progressively greater distances therefrom and adjacent the exit opening for defining a passage way for the ions, said group including an electrode positioned next adjacent said chamber comprised of a pair of elements electrically insulated and maintained at slightly different potentials to provide proper focusing, said group of electrodes being maintained at small fixed potentials for removing ions through said opening, a second group of plates each spaced progressively further from the chamber and having slots therein aligned with said passage way, said group of plates being maintained at potential differences with respect to the walls of said chamber corresponding in magnitude to their distances from the chamber, whereby to provide a substantially uniform potential gradient thereacross for focusing and accelerating the ions into a beam.

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