

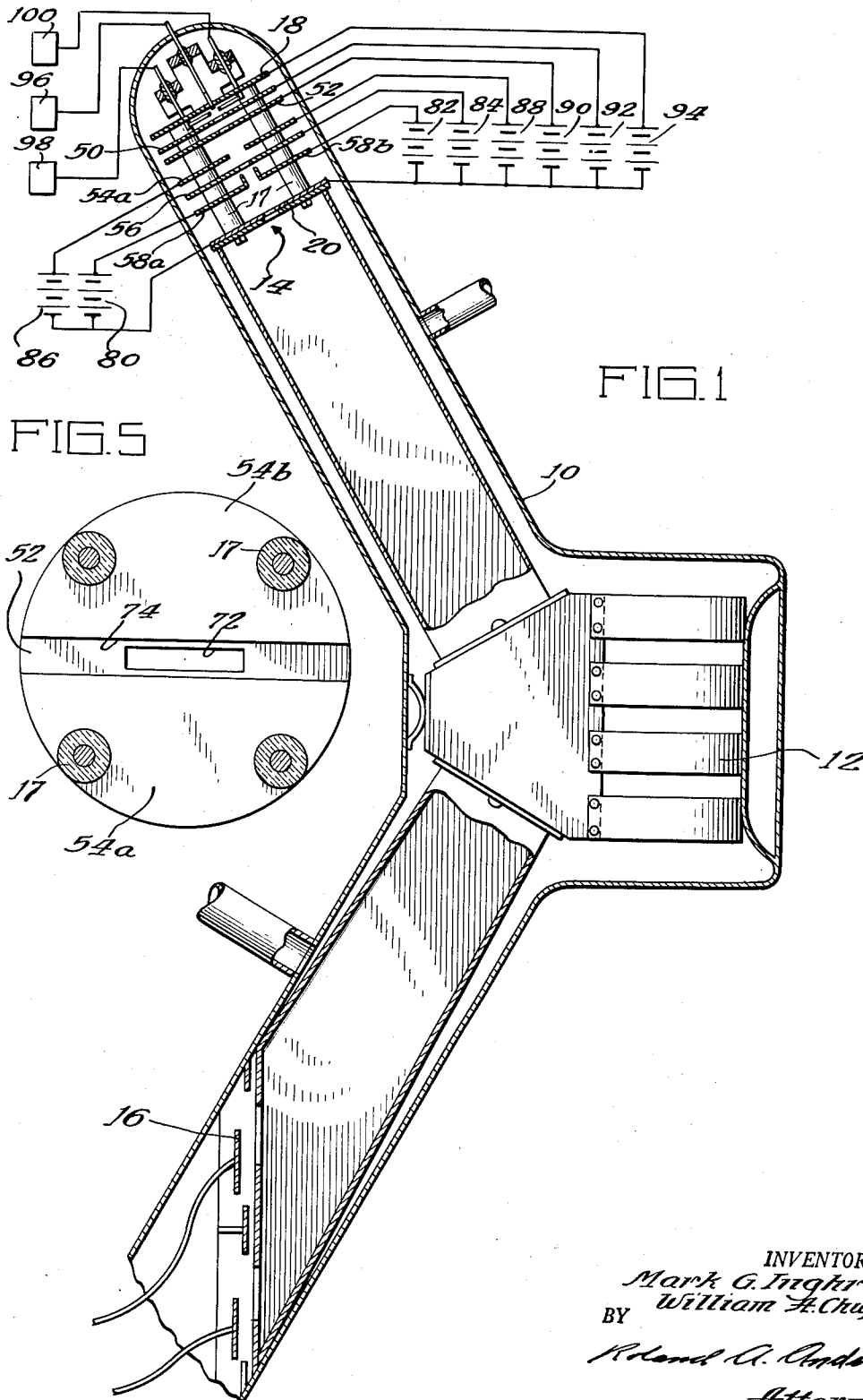
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M. G. INGRAM ET AL
IONIZATION SOURCE

2,733,343

Filed Nov. 24, 1955

2 Sheets-Sheet 1



INVENTORS
Mark G. Ingram
William H. Chupka
BY
Roland A. Anderson
Attorney

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FIG. 2

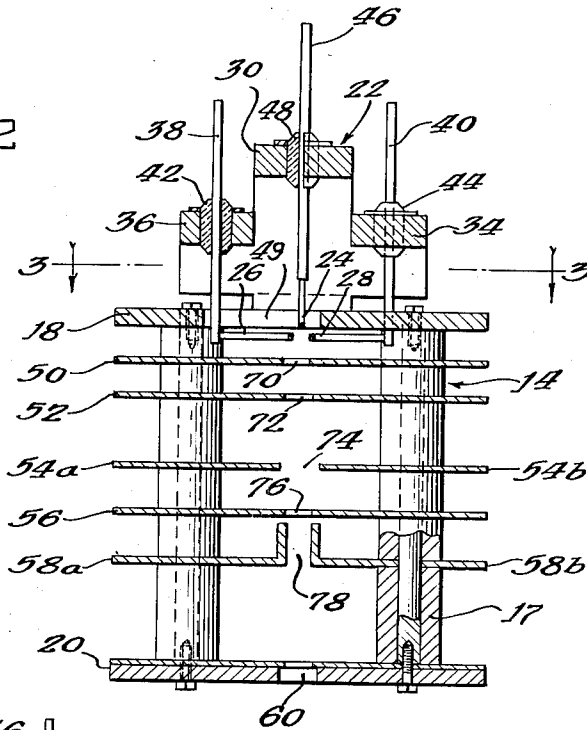


FIG. 4

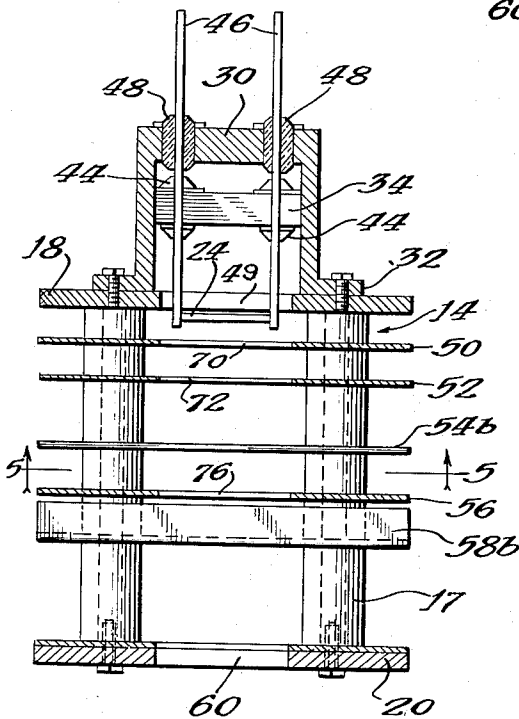
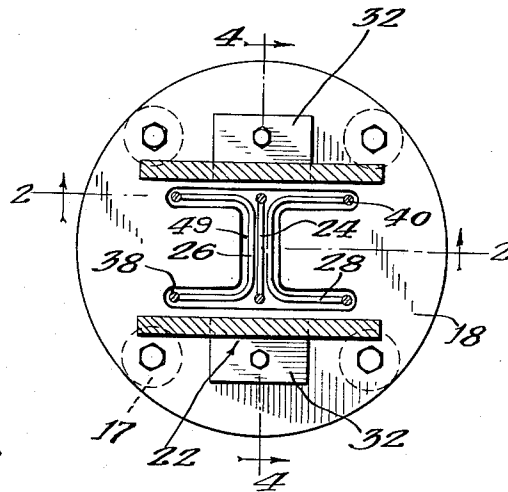


FIG. 3



INVENTORS
Mark G. Ingram
William H. Chupka
BY
Roland A. Anderson
Attorney

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IONIZATION SOURCE

Mark G. Inghram, Chicago, Ill., and William A. Chupka, Cambridge, Mass., assignors to the United States of America as represented by the United States Atomic Energy Commission

Application November 24, 1953, Serial No. 394,521

8 Claims. (Cl. 250—41.9)

The present invention relates to mass spectrometers, and particularly to sources of positive ions suitable for use in mass spectrometers.

The use of surface ionization sources, as distinguished from electron bombardment sources and arc discharge ion sources, has greatly increased in recent years. In simplest form, a surface ionization source consists of a single filament on to which an aliquote of the sample to be analyzed is placed, and by proper selection of the temperature of the filament, molecules and atoms are evaporated as ions from the source. The efficiency of ionization of such a source is given by the following equation:

$$\frac{n^+}{n^0} = e^{\left(\frac{\epsilon(\phi - I)}{KT}\right)}$$

where

$$\frac{n^+}{n^0}$$

is the ratio of the charged to the uncharged component, e is the electronic charge, ϕ is the work function of the evaporating surface, I is the ionization potential of the evaporating component, K is the Boltzmann constant, and T is the absolute temperature. Such an ion source is disclosed in the patent application of Mark G. Inghram and David C. Hess, Serial No. 306,844, filed August 28, 1952, entitled "Ion Source," now Patent No. 2,710,354, issued June 7, 1955.

Several difficulties have been encountered in the use of surface ionization sources, such as described above. First, such surface ionization sources exhibit low ionization efficiency for substances which have ionization potentials which are greater than the work function of the evaporating surface and which evaporate at low temperatures. Low ionization efficiency results under these conditions because the evaporation rate of the material and the temperature at which evaporation occurs cannot be varied independently. The second difficulty encountered with such surface ionization sources is that the molecular form of the evaporated constituent cannot be controlled, since the molecular form is determined essentially only by the temperature in this type of source and the temperature must generally be selected to give the maximum evaporation rate. A third difficulty with surface ionization sources of this type is that relatively high backgrounds occur by surface ionization of hydrocarbons where the aliquot evaporates at a low temperature. It is thus clear that the interdependence of the temperature and evaporation rate in such surface ionization sources is a deterrent to their use.

The present invention provides a surface ionization source in which the evaporation rate of the material to be analyzed is independent of the temperature at which the material is evaporated, thereby alleviating the difficulties which have heretofore limited the use of such surface ionization sources. The present invention therefore provides an ion source which will produce ionized atoms of elements which heretofore have been ionized by

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surface ionization only in the form of molecules. The man skilled in the art will readily perceive many other uses and advantages of the present invention. A more complete understanding of the present invention may be had from a further reading of the disclosure, particularly when viewed in the light of the drawings, in which:

Figure 1 is a sectional view of a mass spectrometer incorporating a surface ionization source constructed according to the teachings of the present invention;

Figure 2 is a sectional view of the surface ionization source shown in Figure 1 taken along the line 2—2 indicated in Figure 3;

Figure 3 is a sectional view taken along line 3—3 of Figure 2;

Figure 4 is a vertical sectional view through the ion source taken along the line 4—4 of Figure 3; and

Figure 5 is a sectional view taken along line 5—5 of Figure 4.

As illustrated in Figure 1, the mass spectrometer is provided with a casing 10 in which a vacuum is maintained. The casing 10 is generally Y-shaped and is provided with a magnet 12 at the junction of the two legs of the Y. An ion source 14, which will be described in detail hereafter, is disposed within one of the legs of the casing 10 near the end thereof, and a collector 16 is disposed adjacent to the end of the other leg of the casing 10. Mass spectrometers of the type here generally described are well known in the art, the copending patent application of Alfred O. C. Nier and Mark G. Inghram, Serial No. 554,965, filed September 20, 1944, entitled "Mass Spectrometer," now Patent Number 2,551,544, issued May 1, 1951, discloses in detail a mass spectrometer of the type generally shown in Figure 1.

The ion source 14, as illustrated in detail in Figures 2 through 5, is provided with four parallel rectangularly positioned posts 17 which support the other elements of the ion source. The posts 17 extend between a filament shield plate 18 and a collimating slit plate 20. A filament supporting member 22 is attached to the filament shield plate 18 and supports an ionization filament 24 and two evaporation filaments 26 and 28.

The filament support member 22 has a generally U-shaped central portion 30 which is provided with flanges 32 to facilitate attaching the member 22 to the filament shield plate 18. Two rectangular bars 34 and 36 are attached to the supporting member 22 and span the open portion of the central portion 30 thereof parallel to the filament shield plate 18. A pair of electrically conducting posts 38 extend through the bar 36 and are insulated therefrom by insulators 42, the evaporation filament 26 being mechanically and electrically connected to the posts 38. In like manner, a pair of electrically conducting posts 40 extend through the bar 34 and are insulated therefrom by insulator 44, and support the evaporation filament 28. The ionization filament 24 is likewise electrically connected to a pair of supporting posts 46 which extend through insulators 48 in the central portion 30 of the filament support 22.

The filament shield plate 18 is provided with an aperture 49 which is generally shaped in the form of an H. The ionization filament 24 is positioned in the cross bar of the H and centrally thereof. The two evaporation filaments 26 and 28 are mounted parallel to the ionization filament 24 and extend from the cross bar of the H along the legs thereof to their respective posts 38 and 40. The evaporation filaments 26 and 28 are also positioned on the side of the filament shield plate 18 opposite to the support member 22, so that the evaporation filaments 26 and 28 are positioned between the ionization filament 24 and the collimating slit plate 20.

A number of slit plates are mounted between the filament shield plate 18 and the collimating slit plate 20, these

are in order of position from the filament shield plate 18, a defocussing slit plate 50, a discriminating slit plate 52, a pair of focussing slit plates 54a and 54b, a collimating slit plate 56, and a pair of beam centering plates 58a and 58b. The collimating slit plate 20 is provided with a slit 60 disposed centrally thereof and generally aligned with the cross bar of the H-shaped aperture 49 in the filament shield plate 18. Each of the plates 50, 52, and 56, which are disposed between the filament shield plate 18 and the collimating slit plate 20, are provided with slits 70, 72 and 76, respectively, which are aligned with the cross bar of the H-shaped aperture 49 in the filament shield plate 18 and the slit 60 of the collimating slit plate 20. Plates 54a and 54b are spaced from each other forming a slit 74 therebetween, and beam centering plates 58a and 58b are also spaced to form slit 78, slits 74 and 78 aligning with the other slits 70, 72, 76 and 60.

In a particular construction of the surface ionization source which will be described throughout this disclosure, the filaments 24, 26 and 28 are constructed in the form of flat ribbons of tungsten, the ribbon being 0.001 by 0.03 inch. The evaporation filaments 26 and 28 are disposed as close to the ionization filament 24 as possible, the two evaporation filaments 26 and 28 being mounted between the ionization filament 24 and the defocussing slit 70, so that the materials evaporated from the filaments 26 and 28 will fall upon the surface of the ionization filament 24 which confronts the slits 70, 72, 74, 76, 78 and 60. In this construction, the evaporation filaments 26 and 28 are spaced from the filament 24 by gaps of not more than 0.01 inch, and in any construction the evaporation filament should not be spaced from the ionization filament by an average gap exceeding twice the width of the ionization filament confronting the evaporation filament.

The length of the cross bar of the H-shaped aperture 49 is approximately three-eighths of an inch, while the total length of the two legs of the H-shaped aperture 49 is approximately five-eighths of an inch, the diameter of the filament shield plate 18, the plates 50, 52, 56 and 20 being the combined halves of plates 54a and 54b, and 58a and 58b being approximately one and one-half inches each. The defocussing slit plate 50 is positioned approximately 0.120 inch from the filament shield plate 18; the discriminating slit plate 52 is positioned approximately 0.120 inch from the defocussing slit plate 50; the focussing slit plates 54a and 54b are positioned approximately 0.240 inch from the discriminating slit plate 52; the collimating slit plate 56 is positioned approximately 0.160 inch from the focussing plates 54a and 54b; the beam centering plates 58a and 58b are positioned approximately 0.160 inch from the collimating slit plate 56; and the collimating slit plate 20 is positioned approximately 0.320 inch from the beam centering plates 58a and 58b. The aperture 49 in the filament shield plate 18 which is in the form of an H is approximately one-eighth inch wide for both the cross arm of the H and the two legs thereof; the slit 70 in the defocussing slit plate 50 is approximately 0.060 inch; the slit 72 in the discriminating plate 52 is approximately 0.030 inch; the slit 74 between the focussing slit plates 54a and 54b is approximately 0.080 inch; the slit 76 in the collimating slit plate 56 is approximately 0.008 inch; the slit 78 separating beam centering plates 58a and 58b is approximately 0.008 inch; and the slit 60 in the collimating slit plate 20 is also approximately 0.008 inch.

The above dimensions are suitable for use with the following voltages: collimating slit plate 20, zero volts; beam centering plates 58a and 58b average approximately +1000 volts, the voltage on these plates being adjustable for purposes of centering the ion beam; the collimating slit plate 56, +1000 volts; the focussing slit plates 54a and 54b average +3500 volts, the potentials on these plates again being varied relative to each other for centering purposes; the discriminating slit plate 52, +4580 volts; the

defocussing slit plate 50, +4980 volts; and the filament shield plate 18, +5000 volts.

The temperatures at which the filaments operate are also important to the production of ions. With the above described ion source, evaporation filaments 26 and 28 may be coated with gadolinium oxide (Gd_2O_3). Evaporation filaments 26 and 28 are then operated at a temperature of 1250° K., and ionization filament 24 is operated at 2500° K. With this construction, it has been found that the ratio of the positively charged components evaporated from this source to the uncharged component is about 3 times 10^{-5} , whereas a single filament surface ionization source, such as previously known to the art, produces a ratio of approximately 10^{-9} . Also, such a single filament surface ionization source produces gadolinium ions mostly as GdO^+ , while the multiple filament ionization source here disclosed produces gadolinium ions in the form Gd^+ .

If uranium in the form of uranium oxide (UO_3) is disposed upon evaporation filaments 26 and 28, and ionization filament 24 is operated at 2700° K. while evaporation filaments 26 and 28 are operated at 1250° K., then the ion source disclosed, voltages and dimensions being maintained the same as previously, will produce U^+ ions. An ion source with a single filament, as previously known, produces uranium ions primarily of the form UO_2^+ . Also, evaporation filaments 26 and 28 may be coated with an oxide of nickel such as NiO , and will produce positive ions of nickel with evaporation filaments 26 and 28 operating at a temperature of 850° K. and the ionization filament operating at a temperature of 2500° K., the voltages and spacings being the same as previously disclosed, where the single filament ionization source is not known to produce positive ions of nickel except due to tertiary emissions.

As shown in Figure 1, the slit plates of the ion source are maintained at the proper voltages by means of conventional power sources, illustrated as batteries. Batteries 80 and 82 are connected between the collimating slit plate 20 and the beam centering plates 58a and 58b, respectively. Battery 84 is connected between the collimating slit plate 20 and the collimating slit plate 56, batteries 86 and 88 are connected between the collimating slit plate 20 and the focussing slit plates 54a and 54b, respectively. Battery 90 is connected between the collimating slit plate 20 and the discriminating slit plate 52. Battery 92 is connected between the collimating slit plate 20 and the defocussing slit plate 50. Also, battery 94 is connected between the collimating slit plate 20 and the filament shield plate 18. It has been found that a potential of not more than 200 volts negative relative to the filament shield plate 18 must be applied to the defocussing slit plate 50 and the discriminating slit plate 52 in order to assure collimation of ions ionized by the ionization filament 24 alone. In addition, the batteries 96, 98 and 100 are used to supply the filament currents for filaments 24, 26 and 28, respectively.

In operating the multiple filament surface ionization source here disclosed, the material to be ionized is placed upon evaporation filaments 26 and 28, and the temperature of these filaments is selected to produce the optimum evaporation rate for the material placed thereon. The evaporation filaments 26 and 28 may be operated at whatever temperature is determined to be most efficient for evaporation of the material by limiting the current flowing through the battery-filament circuits. Also, it is not necessary that two filaments be coated with the material to be ionized, since one of the filaments 26 or 28 could be eliminated, the two filaments having been provided primarily for electrical symmetry. In this manner, ions are emitted from the evaporation filaments 26 and 28; however, as stated above, the emitted ions are often in the form of molecules of the metal to be ionized. A proportion of the emitted molecules will strike the ionization filament 24 which is operated at a very much higher temperature. The temperature of the ionization filament 24

is generally as high as possible consistent with reasonable life for the filament 24; it is highly preferable that the ionization filament 24 be operated at a temperature of at least 2500° K. As a result of the high temperature of filament 24, molecules striking the filament 24 will be broken down into ions of the elements themselves. It is thus clear that the efficiency of ionization has been improved over a single filament surface ionization source. The relative efficiency for ionization of the multiple and single filament sources is given by the equation:

$$\frac{n_2 T_2}{n_1 T_1} = e^{\left\{ \frac{e(\Phi - I)}{KT} \left(\frac{1}{T_2} - \frac{1}{T_1} \right) \right\}}$$

where T_1 and T_2 are the temperatures of the ionization filaments in both sources, e is the electronic charge, Φ is the work function of the evaporating surface, I is the ionization potential of the evaporating component, K is the Boltzmann constant.

It is important that neither of the evaporation filaments 26 nor 28 is focussed upon the slit 60 in the collimating slit plate 20, since the evaporation products from these filaments are of the same purity as those from filament 24 and are not to directly constitute the ion beam. For this reason, the defocussing and discriminating slit plates 50 and 52 are provided and are maintained at a potential slightly negative relative to the filament shield plate 18.

From the foregoing disclosure of the invention, the man skilled in the art will readily devise many modifications and applications for the device here disclosed within the scope of the invention. Hence, it is intended that the invention be not limited by any specific illustration set forth herein, but rather only by the appended claims.

What is claimed is:

1. A source of ions comprising, in combination, an ionization filament, an evaporation filament adapted to be coated with the material to be ionized, means to mount said filaments in adjacent relationship, said filaments being spaced by an average distance equal to not more than twice the width of the ionization filament confronting the evaporation filament.

2. A source of ions comprising, in combination, an evacuated housing, an ionization filament, an evaporation filament adapted to be coated with the material to be ionized, means mounting said filaments within the housing in adjacent relationship, said filaments being spaced by an average distance equal to not more than twice the width of the ionization filament confronting the evaporation filament.

3. A source of ions comprising, in combination, an evacuated housing, an ionization filament, an evaporation filament adapted to be coated with the material to be ionized, means mounting said filaments within the housing in adjacent relationship, said filaments being spaced by an average distance equal to not more than twice the width of the ionization filament confronting the evaporation filament, means to operate the evaporation filament at a temperature producing evaporation of the material to be ionized, means to operate the ionization filament at a temperature producing ionization of the molecules of the material to be ionized, and means to impel the ions produced from the ionization filament.

4. A source of ions comprising, in combination, an evacuated housing, an ionization filament, a pair of evaporation filaments adapted to be coated with the material to be ionized, means disposed within the housing to mount said filaments in adjacent relationship, each of the evaporation filaments being spaced from the ionization filament by an average distance equal to not more than twice the width of the confronting ionization filament, means to operate the evaporation filaments at a temperature producing maximum evaporation of the material to be ionized, means to operate the ionization filament at a temperature of at least 2500° K., and means to impel the ions produced from the ionization filament.

5. A source of ions comprising, in combination, an ionization filament, an evaporation filament adapted to be coated with the material to be ionized, means to mount said filaments in adjacent relationship, said filaments being spaced by an average distance equal to not more than twice the width of the ionization filament confronting the evaporation filament, and means to maintain the temperature of the ionization filament above that of the evaporation filament.

6. A source of ions comprising, in combination, an evacuated housing, an ionization filament, an evaporation filament adapted to be coated with the material to be ionized, means mounting said filaments within the housing in adjacent relationship, said filaments being spaced by an average distance equal to not more than twice the width of the ionization filament confronting the evaporation filament, and means to impel the ions produced from the ionization filament including a filament shield plate and at least two slit plates, the filament shield plate having an aperture disposed therein positioned adjacent to the ionization filament, and the other two slit plates having slits aligned with the ionization filament, the slit plate disposed between the filament shield plate and the other slit plate being maintained at a potential not more than 200 volts negative relative to the filament shield plate, and the other slit plate being maintained at a potential more negative than the first slit plate.

7. A source of ions comprising, in combination, an evacuated housing, an ionization filament, an evaporation filament adapted to be coated with the material to be ionized, means mounting said filaments within the housing in adjacent relationship, said filaments being spaced by an average distance equal to not more than twice the width of the ionization filament confronting the evaporation filament, means to operate the evaporation filament at a temperature producing evaporation of the material to be ionized, means to operate the ionization filament at a greater temperature than the evaporation filament, and means to impel the ions produced from the ionization filament including a filament shield plate having an aperture therein, the ionization filament being disposed within the aperture of the filament shield plate, and at least two slit plates, the slit plates having slits aligned with the ionization filament, and means to maintain the slit plate adjacent to the filament shield plate at a potential negative to the filament shield plate by not more than 200 volts, and means to maintain the other slit plate at a potential more negative than the first slit plate relative to the filament shield plate.

8. A source of ions comprising, in combination, an evacuated housing, an ionization filament, a pair of evaporation filaments adapted to be coated with the material to be ionized, means disposed within the housing to mount said filaments in adjacent relationship, each of the evaporation filaments being spaced from the ionization filament by an average distance equal to not more than twice the width of the confronting ionization filament, means to operate the evaporation filaments at a temperature producing maximum evaporation of the material to be ionized, means to operate the ionization filaments at a temperature of at least 2500° K., and means to impel the ions produced from the ionization filament including a filament shield plate having an aperture therein including a portion forming a slit, the ionization filament being disposed within said slit portion and the evaporation filaments being disposed adjacent thereto, a defocussing slit plate disposed adjacent to the filament shield plate, a discriminating slit plate disposed adjacent to the defocussing slit plate, a pair of focussing slit plates disposed adjacent to the discriminating slit plate, a collimating slit plate disposed adjacent to the defocussing slit plates, a pair of beam centering plates disposed adjacent to the collimating slit plate, and a second collimating slit plate disposed adjacent to the beam centering plates, the defocussing slit plate, discriminating slit plate,

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collimating slit plate, and second collimating slit plate being provided with aligned slits and the focussing slit plates and beam centering slit plates being spaced from each other to form slits aligned with the slits in the slit plates, means to apply a large positive potential on the filament shield plate relative to the second collimating

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slit plate, and means to apply a potential of not more than 200 volts negative with respect to the filament shield plate on the defocussing slit plate and on the discriminating slit plate.

No references cited