

May 14, 1957

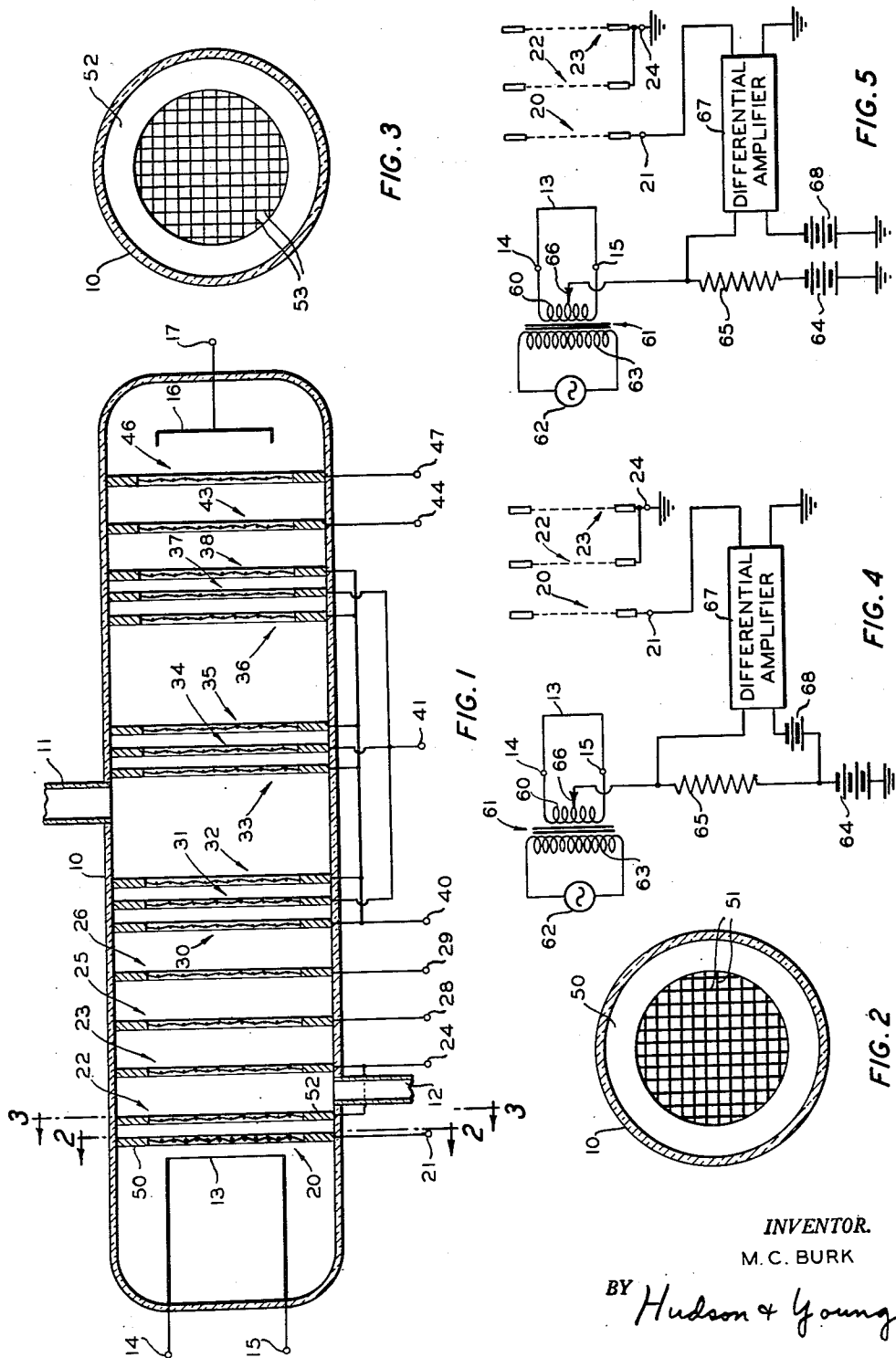
M. C. BURK

2,792,500

ION SOURCE

Filed Feb. 26, 1954

3 Sheets-Sheet 1



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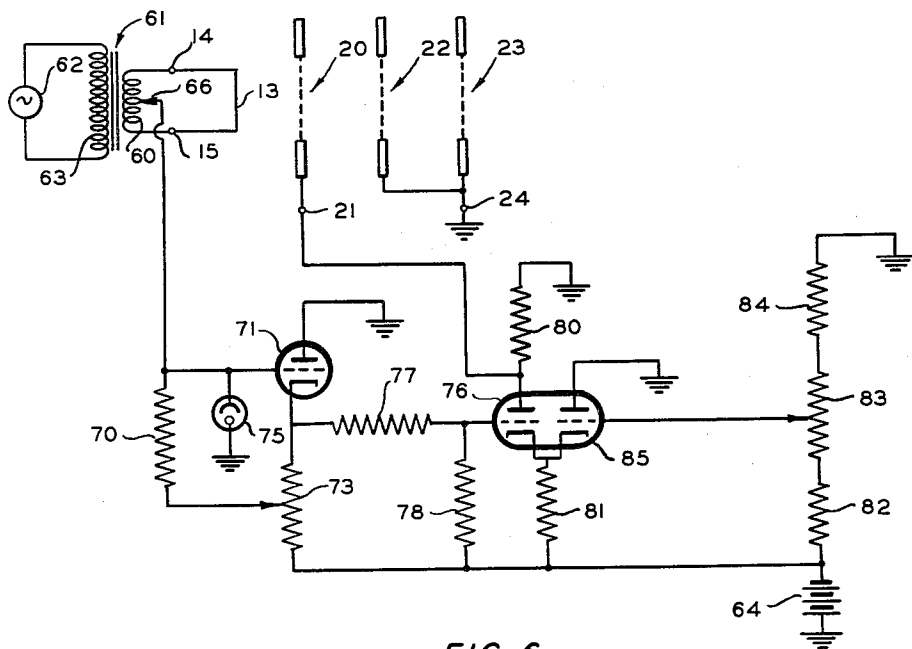


FIG. 6

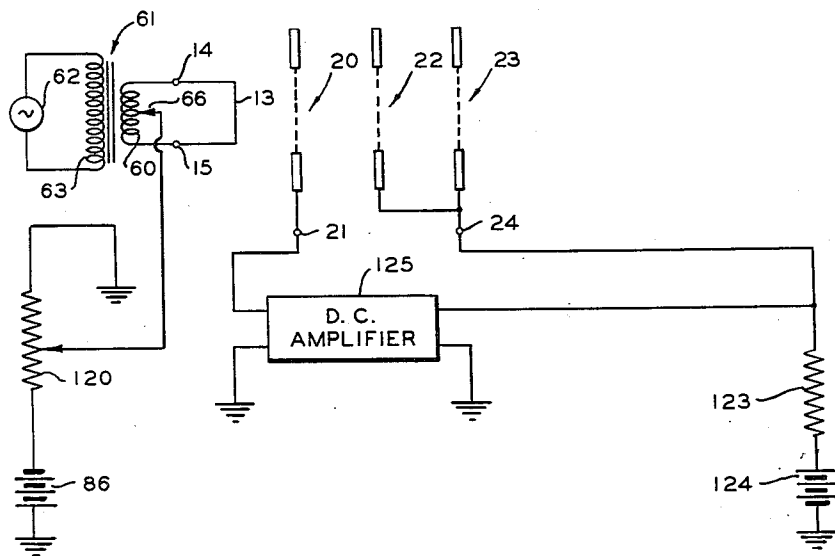


FIG. 8

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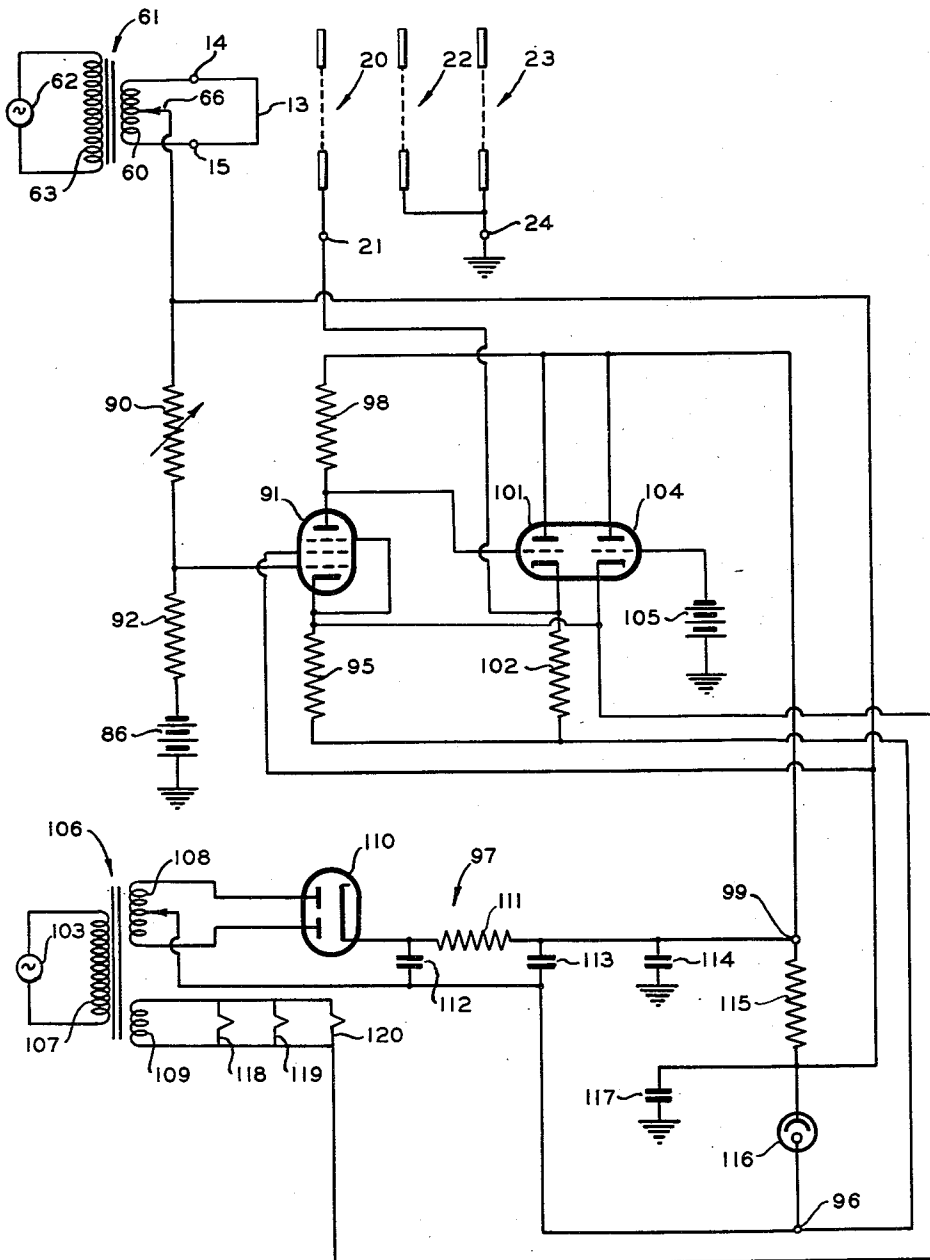


FIG. 7

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ION SOURCE

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9 Claims. (Cl. 250—41.9)

This invention relates to the formation of ions by the bombardment of a gas with a stream of electrons. In another aspect it relates to an electron emission regulator for an ion source.

Mass spectrometry comprises, in general, ionizing a sample of material under investigation and separating the resulting ions according to their individual masses to determine the relative abundance of ions of selected masses. The material to be analyzed usually is provided as a gas, and this gas is bombarded by a stream of electrons to produce the desired ions. The gas molecules may be ionized by the removal of electrons, by breaking up the molecules, or both. Although both positive and negative ions may be formed by such electron bombardment, most mass spectrometers make use of only the positive ions. These positive ions are accelerated out of the region of the electron beam by means of a negative potential which imparts equal kinetic energies to ions having like charges such that ions of different masses have different velocities after passing through the electrical field and consequently have different momenta. The presently known mass spectrometers can be classified in one of two general groups: the momentum and velocity selection types. The momentum selection instruments sort the ions into beams of different masses by means of a magnetic and/or an electrical deflecting field. Ions of a selected mass are allowed to impinge upon a collector plate to which is connected a suitable indicating meter. The velocity selection instrument sort the ions according to their velocities, and are referred to generally as time-of-flight mass spectrometers.

In all these mass spectrometers it is common practice to produce the ions by bombardment of molecules with a stream of electrons. The electrons generally are emitted from a heated filament and are accelerated by an electrical field into an ionization chamber which contains the gas molecules to be ionized. It is, of course, important to maintain the electron beam of constant magnitude because variations in the electron emission tend to introduce corresponding variations in the degree of ionization. This in turn introduces errors in the analysis. The filament normally is heated by an electrical current and some degree of control of the electron emission can be maintained by regulating the current supplied to the filament. However, even small variations in the current supplied to the filament results in changes in the electron emission. Furthermore, this control procedure does not compensate for changes in emission which may be due to other factors such as changes in the emitting properties of the filament.

In accordance with the present invention the electron emission is held constant by means of an electrical field disposed adjacent the filament in the path of the electron beam. This field, which can be either positive or negative with respect to the potential on the filament, is conveniently established by means of a screen electrode which can be a wire mesh mounted generally perpendicular to the path of the electron beam. By varying the

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potential on this screen electrode, the flow of electrons therethrough can be regulated in a precise manner. Circuit means are provided to establish a first voltage proportional to the magnitude of the electron flow. This first voltage is compared with a constant reference voltage, and the difference between the two voltages controls the potential applied to the screen electrode. Thus, if the electron emission from the filament should tend to increase, the potential applied to the grid is made more negative to tend to repel the electrons flowing therethrough into the ionization chamber. Conversely, if the flow of electrons from the filament tends to decrease, the grid is made more positive to tend to increase the flow of electrons into the ionization chamber. By this procedure it is possible to maintain a constant flow of electrons into the ionization chamber, and the flow can be retained constant irrespective of changes in heat supplied to the filament or changes in the emitting properties of the filament.

Accordingly, it is an object of this invention to provide means to regulate the production of ions by the bombardment of gases with a stream of electrons.

Another object is to provide an electronic emission regulator to control a beam of electrons in an ion source.

A further object is to provide apparatus for supplying a constant source of ions for analysis by mass spectrometry.

Various other objects, advantages and features of this invention should become apparent from the following detailed description taken in conjunction with the accompanying drawing in which:

Figure 1 is a schematic representation of a mass spectrometer tube having the emission regulating apparatus of the present invention incorporated therein;

Figure 2 is a sectional view taken along line 2—2 in Figure 1;

Figure 3 is a sectional view taken along line 3—3 in Figure 1;

Figure 4 is a schematic circuit diagram of a first embodiment of the emission regulator;

Figure 5 is a schematic circuit diagram of a second embodiment of the emission regulator;

Figure 6 is a detailed circuit diagram of an embodiment of the emission regulator;

Figure 7 is a detailed circuit diagram of an embodiment of the emission regulator; and

Figure 8 is a schematic circuit diagram of still another embodiment of the emission regulator.

Referring now to the drawing in detail and to Figure 1 in particular, there is shown a mass spectrometer tube which is enclosed within a vessel 10. The interior of vessel 10 is maintained at a reduced pressure by means of a vacuum pump, not shown, which is connected to a conduit 11 which communicates with vessel 10; and a second conduit 12 communicates with vessel 10 to supply a gas sample to be analyzed. A filament 13 is mounted at one end of vessel 10. Electrical terminals 14 and 15 are connected to the respective ends of filament 13 to supply heating current thereto. At the opposite end of vessel 10 there is positioned an ion collector 16 which is connected to an external terminal 17. A detector, not shown, is connected to terminal 17. A series of grids is positioned between filament 13 and collector 16. Proceeding from filament 13 toward collector 16, these grids comprise a screen electrode 20 which is employed to control the electron flow from filament 13. Electrode 20 is connected to a terminal 21. A pair of grids 22 and 23 defines an ionization chamber into which the gas sample from conduit 12 is directed. Grids 22 and 23 are electrically connected to one another and to a terminal 24 which is maintained more positive than

filament 13. The next two grids 25 and 26 are employed to focus the resulting ions in a beam which is directed toward collector 16. Grids 25 and 26 are connected to respective terminals 28 and 29 which are maintained at negative potentials. The next three grids 30, 31 and 32 are spaced relatively close to one another, and a corresponding series of grids 33, 34 and 35 is spaced from this first group. A third series of grids 36, 37 and 38 is spaced from the second set. Grids 30, 32, 33, 35, 36 and 38 are electrically connected to one another and to a terminal 40. Grids 31, 34 and 37 are electrically connected to one another and to a terminal 41. A negative potential is applied to terminal 40 and an alternating potential is applied between grids 40 and 41. The next grid 43 is connected to a terminal 44 which has a positive potential applied thereto, and the final grid 46 is connected to a terminal 47 which has a negative potential applied thereto.

Electrons emitted from filament 13 are accelerated into the ionization chamber between grids 22 and 23 wherein they bombard the gas molecules to provide ions. The resulting positive ions are accelerated out of the ionization chamber by the negative potentials on grids 25 and 26 and are directed toward collector 16. The next nine grids 30—38 function to accelerate the ions by amounts which are functions of the masses of and the charges on the individual ions. Ions of a predetermined mass receive sufficient energy to overcome the positive retarding potential applied to grid 43 and are thus able to reach collector 16. The purpose of grid 46 is to repel any secondary electrons which may be formed within the tube. For a more detailed description of the operation of the accelerating grids 30—38, reference is made to U. S. Patent 2,535,032.

In one embodiment of this invention, filament 13 was formed of a tungsten wire approximately one inch long and 0.007 inch in diameter. Screen electrode 20, which is shown in detail in Figure 2, was positioned approximately $\frac{1}{8}$ inch from filament 13. Electrode 20 was in the form of a wire mesh contained within an annular member 50. The individual wires 51 of the mesh were formed of tungsten wire having a diameter of approximately 0.010 inch. Adjacent parallel wires 51 were spaced approximately $\frac{1}{10}$ inch from one another. The first grid 22 of the ionization chamber was spaced approximately $\frac{1}{8}$ inch from grid 20 and was formed of a wire mesh contained within an annular ring 52. Wires 53 forming the mesh of grid 22 were of tungsten wire having a diameter of approximately 0.001 inch. Adjacent parallel wires 53 were spaced approximately 0.020 inch from one another.

In Figure 4 there is shown a schematic circuit diagram of one embodiment of the electron emission regulator of this invention. Terminals 14 and 15 are connected to the respective end terminals of the secondary winding 60 of a voltage step-down transformer 61. A source of alternating potential 62 is applied across the primary winding 63 of transformer 61. The center tap 66 of secondary winding 60 is connected to the negative terminal of a voltage source 64 through a resistor 65. Center tap 66 is also connected to one input terminal of a differential amplifier 67. The second input terminal of amplifier 67 is connected to the negative terminal of a voltage source 68, the positive terminal of voltage source 68 being connected to the negative terminal of voltage source 64. One output terminal of amplifier 67 is connected to grid 20 and the second output terminal is grounded. Amplifier 67 is adapted to provide an output voltage of magnitude proportional to the difference in voltages applied to the input terminals thereof.

The electrons emitted from heated filament 13 are supplied by voltage source 64 and flow through resistor 65. The potential at the center tap 66 of transformer winding 60 obviously is a function of the current flow

through resistor 65. If the ion emission current should tend to increase, the potential at center tap 66 becomes more positive due to the increased voltage drop across resistor 65. Under this condition, the output voltage of amplifier 67 decreases such that screen electrode 20 becomes more negative. This decrease in screen electrode potential tends to retard the electron flow from filament 13 toward grounded grids 22 and 23 such that the electron flow into the ionization chamber is reduced to compensate for the original increase. Conversely, if the electron flow from filament 13 should tend to decrease, then the potential at center tap 66 is decreased such that output potential from amplifier 70 becomes more positive to increase the electron flow between filament 13 and grids 22 and 23.

The circuit shown in Figure 5 is generally similar to that shown in Figure 4 except that the positive terminal of voltage source 68 is connected to ground rather than to the negative terminal of voltage source 64. The operation of the circuit of Figure 5 is substantially the same as that of the circuit of Figure 4, the only difference being that the reference potential supplied by voltage source 68 is independent of the circuit including resistor 65 and voltage source 64.

In Figure 6 there is shown a detailed circuit which has been employed as amplifier 67 in Figure 4. The center tap 66 of transformer winding 60 is connected to the first terminal of a resistor 70 and to the control grid of a triode 71. The anode of triode 71 is grounded and the cathode thereof is connected to the negative terminal of voltage source 64 through a potentiometer 73. The second end terminal of resistor 70 is connected to the contactor of potentiometer 73. A voltage limiting tube 75 is connected between the control grid of triode 71 and ground. The cathode of triode 71 is connected to the control grid of a second triode 76 through a resistor 77, and the control grid of triode 76 is connected to the negative terminal of voltage source 64 through a resistor 78. The anode of triode 76 is connected to ground through a resistor 80 and directly to screen electrode 20. The cathode of triode 76 is connected to the negative terminal of voltage source 64 through a resistor 81. The negative terminal of voltage source 64 is connected to ground through a voltage dividing network comprising a resistor 82, a potentiometer 83 and a resistor 84. The contactor of potentiometer 83 is connected to the control grid of a third triode 85. The anode of triode 85 is grounded and the cathode of triode 85 is connected to the cathode of triode 76. The filaments of triodes 71, 76 and 85 have been omitted from Figure 6 for purposes of simplicity.

The current flow through cathode resistor 81 is divided between triodes 76 and 85 in accordance with the potentials applied to the respective control grids and anodes of these two triodes. The potential applied to screen electrode 20 is a function of the current flow through triode 76, which results in a voltage drop across resistor 80. The potential on the control grid of triode 85 is maintained at a reference value by the setting of the contactor of potentiometer 83. The potential on the control grid of triode 76, however, is a function of the potential at center tap 66 of transformer winding 60, which in turn is a function of the current flow through resistor 70. If the electron emission from filament 13 should tend to increase, the potential applied to the control grid of triode 71 is increased because of the increased current flow through resistor 70 which results in an increased voltage drop thereacross. The increase in potential applied to the control grid of triode 71 results in increased current flow through that tube and in a corresponding increase in the potential at the cathode thereof. This in turn increases the potential at the control grid of triode 76 so as to increase the current flow through triode 76 and resistor 80. The increased current flow through resistor 80 results in the potential at the anode of triode

76 and the potential at screen electrode 20 being decreased. If the current flow from filament 13 should tend to decrease, the above-mentioned potential changes are reversed such that the potential at grid 20 is increased. The function of voltage limiting tube 75 is to prevent an excess rise in voltage on the control grid of triode 71 in the event of a power failure in the filament heater circuit. It should thus be apparent that the circuit of Figure 6 operates to maintain a constant flow of electrons into the ionization chamber irrespective of changes in electron emission from filament 13. This circuit normally is operated such that grid 20 is maintained at a potential more positive than the potential at filament 13.

In one embodiment of this invention the following circuit values were employed: resistor 70, 270,000 ohms; potentiometer 73, 300,000 ohms; resistor 77, 180,000 ohms; resistor 78, one megohm; resistor 81, 110,000 ohms; resistor 84, 120,000 ohms; resistor 82, 375,000 ohms; potentiometer 83, 70,000 ohms; voltage source 64, 600 volts; the voltage across transformer winding 60, 4 to 5 volts; triode 71, type 6C4; triodes 76 and 85, type 12AU7; and tube 75, type 0A2.

In Figure 7 there is shown a second embodiment of the emission regulator which can be employed to advantage when it is desired to increase the electron emission current. Screen electrode 20 is maintained at a potential more positive than the potential at filament 13. Under these conditions, provision must be made to compensate for any resulting screen electrode current. The center tap 65 of transformer winding 60 is connected to one end terminal of a variable resistor 90, and the second end terminal of resistor 90 is connected to the control grid of a pentode 91 and to the first end terminal of a resistor 92. The second end terminal of resistor 92 is connected to the negative terminal of a voltage source 86, the positive terminal of which is grounded. The cathode of pentode 91 is connected through a resistor 95 to the negative terminal 96 of an isolated power supply unit 97 which is described in detail hereinafter. The anode of pentode 91 is connected through a resistor 98 to the positive terminal 99 of power supply circuit 97. The anode of pentode 91 is also connected to the control grid of a triode 101. The anode of triode 101 is connected to positive terminal 99, and the cathode of triode 101 is connected to negative potential terminal 96 through a resistor 102. The cathode of triode 101 is also connected to grid 20. A second triode 104 is provided with its anode connected to positive terminal 99. The control grid of triode 104 is connected to the negative terminal of a constant voltage source 105, the positive terminal of source 105 being grounded. The cathode of triode 104 is connected to the cathode of pentode 91.

Power supply circuit 97 includes a transformer 106 having a primary winding 107 and a pair of secondary windings 108 and 109. A source of alternating potential 103 is applied across winding 107. The end terminals of transformer winding 108 are connected to the two anodes, respectively, of a double diode 110. The cathode of diode 110 is connected to positive potential terminal 99 through a resistor 111. The center tap of transformer winding 108 is connected to negative potential terminal 96. A first capacitor 112 is connected between the cathode of double diode 110 and terminal 96; a second capacitor 113 is connected between terminals 99 and 96; and a third capacitor 114 is connected between terminal 99 and ground. A resistor 115 and a voltage regulating tube 116 are connected in series relation with one another between terminals 99 and 96. The junction between resistor 115 and tube 116 is connected directly to the screen grid of pentode 91 and to the center tap 65 of transformer winding 60. This junction is also connected to ground through a capacitor 117. Transformer winding 109 provides current for the filaments 118, 119 and 120 of vacuum tubes 98, 101 and

104, and 110, respectively. One end terminal of transformer winding 109 is connected to the cathodes of triodes 104 and pentode 91.

The emission regulator circuit of Figure 7 operates in substantially the same manner as the circuit in Figure 6. The current flow through triode 104 is maintained substantially constant because of the constant potential applied to the control grid thereof from voltage source 105. The electron flow through triode 101, however, is a function of the electron emission from filament 13. If the electron emission from filament 13 should increase, there is an increased current flow through resistors 90 and 92 such that the potential at the junction between these resistors tends to increase. This potential is applied to the control grid of pentode 91 to increase the current flow through the pentode 91 and resistor 98. This increased current flow through resistor 98 results in the potential at the anode of pentode 91 being lowered, and this lowered potential is in turn applied to the control grid of triode 101 to decrease the current flow through triode 101 and cathode resistor 102. The decrease in current flow through resistor 102 lowers the potential at the cathode of triode 101 and the potential at screen electrode 20 to decrease the electron emission flow. Any decrease in electron emission from filament 13 changes the above-mentioned potentials in the opposite manner so as to tend to increase the electron flow through screen electrode 20. In order to eliminate the effect of any screen electrode current, a direct circuit path is provided between screen electrode 20 and filament 13 through resistor 102, tube 116 and transformer winding 60.

In one embodiment of this invention excellent results were obtained with the circuit of Figure 7 having the following component values: resistor 90, 25,000 ohms; resistor 92, 250,000 ohms; resistor 98, 220,000 ohms; resistor 95, 6800 ohms; resistor 102, 56,000 ohms; resistor 111, 1,500 ohms; resistor 115, 10,000 ohms; capacitor 112, 20 microfarads; capacitor 113, 40 microfarads; capacitor 114, 0.05 microfarad; capacitor 117, 8 microfarads; voltage source 93, 600 volts; voltage source 105, 100 volts; pentode 91, type 6AU6; triodes 101 and 104, type 12AT7; tube 116, type 0B2; and double diode 110, type 6X4.

In Figure 8 there is shown still another embodiment of the emission regulator. The center tap 66 of transformer winding 60 is connected to the contactor of a potentiometer 120. One end terminal of potentiometer 120 is grounded and the second end terminal is connected to the negative terminal of a voltage source 86, the positive terminal of which is grounded. Grids 22 and 23, which form the ionization chamber, are connected to one terminal of a resistor 123. The second terminal of resistor 123 is connected to the positive terminal of a voltage source 124, the negative terminal of which is grounded. Grids 22 and 23 also are connected to one input terminal of a direct current amplifier 125, the second input terminal of which is grounded. Amplifier 125 is designed to provide an output signal in phase with the input signal. The first output terminal of amplifier 125 is connected to screen electrode 20 and the second output terminal of amplifier 125 is grounded. If the electron flow from filament 13 to the ionization chamber should tend to increase, there is an increased flow through resistor 123 such that grids 22 and 23 become more negative. This potential decrease is applied to amplifier 125 and the amplified output signal therefrom is applied to screen electrode 20 as previously described to decrease the potential at grid 22 to decrease electron flow therethrough. The circuit of Figure 8 thus tends to regulate the electron emission in terms of the number of electrons impinging grids 22 and 23 of the ionization chamber.

From the foregoing description it should be apparent that there is provided in accordance with this invention an improved electron emission regulator which is particularly adapted for use in the ion source of a mass spec-

trometer tube. This system operates with a minimum expenditure of power and the response time is almost instantaneous since it is in no way limited by thermal inertia which occurs in regulators which function to maintain the filament temperature constant. Because of the rapid response, this regulator is capable of substantially eliminating the 60 cycle ripple which may be caused by using an alternating current powered filament. While the emission regulator has been described in conjunction with a particular form of mass spectrometer tube, it should be apparent that the invention is equally adapted to other types of mass spectrometer tubes such as those employing magnetic deflection. Furthermore, this invention is useful whenever it is desired to provide an ion source utilizing an electron beam of constant magnitude.

While the invention has been described in conjunction with present preferred embodiments thereof, it should be apparent that the invention is not limited thereto. As employed herein, the term "screen electrode" is intended to designate a structure which is substantially open to the flow of electrons therethrough. The wire mesh screen of Figure 2 has been found to give excellent results, although it should be apparent that other screen configurations can be employed.

What is claimed is:

1. An ion source comprising, in combination, an ionization chamber, an electron emitting filament, means for directing electrons emitted from said filament to said ionization chamber, a screen electrode positioned in the path of said electrons, a reference voltage, means to establish a second voltage which is a function of the electron emission from said filament, a differential amplifier, means connecting one input terminal of said amplifier to said reference voltage, means connecting the second input terminal of said amplifier to said second voltage, means connecting one output terminal of said one amplifier to said screen electrode, and means connecting the second output terminal of said amplifier to a point of reference potential, whereby the potential applied to said screen electrode becomes more positive when said electron emission decreases and becomes more negative when said electron emission increases whereby the electron flow into said ionization chamber remains substantially constant.

2. An ion source comprising, in combination, an ionization chamber, an electron emitting filament, a source of first potential, an impedance element connected between said filament and said source of first potential, means for directing electrons emitted from said filament to said ionization chamber, a screen electrode positioned in the path of said electrons, a source of second potential, a differential amplifier, one input terminal of said amplifier being connected to said source of second potential, the second input terminal being connected between said filament and said impedance element, one output terminal of said amplifier being connected to said screen electrode, and the second output terminal of said amplifier being connected to a point of reference potential, whereby the potential applied to said screen electrode becomes more positive when said electron emission decreases and becomes more negative when said electron emission increases whereby the electron flow into said ionization chamber remains substantially constant.

3. The combination in accordance with claim 2 wherein said source of second potential comprises a voltage source, the negative terminal of which is connected to said one input terminal of said amplifier and the positive terminal of which is connected between said impedance element and said source of first potential.

4. The combination in accordance with claim 2 wherein said source of second potential comprises a voltage source, the negative terminal of which is connected to said one input terminal of said amplifier and the positive terminal of which is connected to said point of reference potential.

5. An ion source comprising, in combination, an ionization chamber, an electron emitting filament, a source of

first potential, an impedance element connected between said filament and said source of first potential, means for directing electrons emitted from said filament to said ionization chamber, a screen electrode positioned in the path of said electrons, a first electron tube having at least a cathode, an anode and a control grid, a second electron tube having at least an anode, a cathode and a control grid, the cathode circuits of said first and second tubes having a common portion, means to apply a predetermined potential to the grid of said first tube, means coupling the control grid of said second tube to the junction between said filament and said impedance element whereby the potential applied to the control grid of said second tube is a function of the electron emission from said filament, and means connecting the output signal from said second tube to said screen electrode whereby the potential applied to said screen electrode becomes more positive when said electron emission decreases and becomes more negative when said electron emission increases whereby the electron flow into said ionization chamber remains substantially constant.

6. An ion source comprising, in combination, an ionization chamber, an electron emitting filament, a source of first potential, an impedance element connected between said filament and said source of first potential, means for directing electrons emitted from said filament to said ionization chamber, a screen electrode positioned in the path of said electrons, a first electron tube having at least a cathode, an anode and a control grid, the anode of said first tube being connected to a point of reference potential, the control grid of said first tube being connected to the junction between said filament and said impedance element, a first resistor connected between the cathode of said first tube and said source of first potential, second and third electron tubes each having at least an anode, a cathode and a control grid, a second resistor having one end terminal connected to said source of first potential, the second end terminal of said second resistor being connected to the cathodes of said second and third tubes, the anode of said second tube being connected to said point of reference potential, a third resistor connected between the anode of said third tube and said point of reference potential, a potentiometer having one end terminal connected to said source of first potential and the second end terminal connected to said point of reference potential, the contactor of said potentiometer being connected to the control grid of said second tube, and circuit means connecting the cathode of said first tube to the control grid of said third tube, the anode of said third tube being connected to said screen electrode whereby the potential applied to said screen electrode becomes more positive when said electron emission decreases and becomes more negative when said electron emission increases whereby the electron flow into said ionization chamber remains substantially constant.

7. The combination in accordance with claim 6 further comprising voltage limiting means connected between the control grid of said first tube and said point of reference potential.

8. An ion source comprising, in combination, an ionization chamber, an electron emitting filament, a source of first potential, an impedance element connected between said filament and said source of first potential, means for directing electrons emitted from said filament to said ionization chamber, a screen electrode positioned in the path of said electrons, a first electron tube having at least a cathode, an anode and a control grid, a source of second potential, a first resistor connected between the anode of said first tube and said source of second potential, the control grid of said first tube being connected to the junction between said filament and said impedance element, a source of third potential which is more negative than said source of second potential, a second resistor connected between the cathode of said first tube and said source of third potential, second and third electron tubes each

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having at least a cathode, an anode and a control grid, the cathode of said second tube being connected to the cathode of said first tube, the anodes of said second and third tubes being connected to said source of second potential, a third resistor connected between the cathode of said third tube and said source of third potential, the anode of said first tube being connected to the control grid of said third tube, and circuit means connecting the cathode of said third tube to said screen electrode whereby the potential applied to said screen electrode becomes more positive when said electron emission decreases and becomes more negative when said electron emission increases whereby the electron flow into said ionization chamber remains substantially constant.

9. An ion source comprising, in combination, an ionization chamber defined by a pair of spaced electrodes, an electron emitting filament, a screen electrode positioned between said filament and said ionization chamber, a

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source of first potential, an impedance element connected between said source of first potential and said pair of spaced electrodes, an amplifier adapted to provide an output signal in phase with the input signal applied thereto, means connecting one input terminal of said amplifier to said pair of spaced electrodes and the other input terminal to a point of reference potential, and means connecting one output terminal of said amplifier to said screen electrode and the other output terminal to a point of reference potential.

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