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POSITIVE AND NEGATIVE ION RECORDING SYSTEM FOR MASS SPECTROMETER

(71) We, THE UNIVERSITY OF VIRGINIA of Charlottesville, Virginia 22901, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:-

The present invention relates generally to the field of quadrupole mass spectroscopy, and more particularly to a method and apparatus for producing and monitoring both positive and negative ions using a quadrupole mass spectrometer.

In quadrupole mass spectrometers, ions of different masses are separated by a quadrupole filter. Although positive and negative ions can be transmitted simultaneously through such a filter, conventionally available devices permit ions of only one polarity to be extracted from the filter for detection and data processing. Normally, only positive ions are detected, primarily because commercially available devices are constructed and operated under conditions favoring the generation of positive ions and because electron multipliers are normally operated at negative potentials, thus tending to attract only positive ions and repel negative ions.

Some devices have been constructed which permit *sequential* detection of positive and negative ions. One such device is sold by Extranuclear Laboratories, Incorporated, of Pittsburgh, Pennsylvania. This device is a quadrupole mass spectrometer which includes a toggle switch for reversing voltage polarities on a single electron multiplier and ion source. A delay of approximately ten seconds is required between recording ions of different polarities. The delay period for switching between positive and negative ion detection in these machines is sufficiently long so that simultaneous or near simultaneous recording of ions of both polarities is completely impossible, with the result that accurate mass measurements can only be made in great difficulty and not at all on certain ions with such machines. Similarly, it is not possible to record both positive and negative ion spectra on a single injection of sample molecules introduced into such machines through a gas chromatograph, for example. These factors emphasize the point that sequential detection of positive and negative ions is not at all equivalent to simultaneous, or effectively simultaneous, detection of both polarities of ions. The capability of sequential detection of both types of ions is essentially equivalent to using two separate mass spectrometers to process positive and negative ions, and fails to attain the synergistic effects possible with simultaneous or near simultaneous detection.

It is well understood by those skilled in the art that substantially simultaneous recording of both positive and negative ion species in quadrupole mass spectrometers would be highly desirable in that it would greatly facilitate the making of accurate mass measurements, among other things. In obtaining mass measurements, for example, it is necessary that some means be found to distinguish the ions emanating from an internal standard from those emanating from an unknown sample of nearly the same unit mass as the standard. According to the present invention, distinguishing between ions emanating from the standard and those emanating from the unknown sample is greatly facilitated by operating the mass spectrometer under conditions such that only negative ions, for example, are generated by the internal standard while only positive ions are generated by the sample. Both types of ions are recorded essentially simultaneously using a pulsed ion source, single quadrupole filter, dual electron multiplier detector and dual channel or stereo recording devices with the result that mass measurements at low ppm levels can be made with and with great simplicity.

According to the present invention a method of generating and monitoring positive and negative ions effectively simultaneously using a quadrupole mass spectrometer comprises operating an ion source of the said mass spectrometer under conditions favouring the generation of positive and negative ions, rapidly switching the potentials applied to repeller, source and lens electrodes of the said quadrupole mass spectrometer ion source between selected positive and negative levels, and separately detecting the said positive and negative ions.

The method may further comprises producing electrical signals representative of the said positive and negative ions detected in the said step of detecting and processing the said electrical signals.

Perfluorokerosene is preferably used as an internal standard material.

Isobutane is preferably used as the reagent gas at a pressure of approximately one torr.

The potentials applied to the said repeller, source and lens electrodes are preferably changed at a frequency in the range between 1 and 100 kHz, e.g. approximately 10 kHz.

The invention also extends to apparatus for enabling a quadrupole mass spectrometer having an ion source including repeller, source and lens electrodes to effectively simultaneously generate and monitor both positive and negative ions, which comprises high frequency switching means for alternately applying selected positive and negative potentials to the said repeller, source and lens electrodes, and dual detector means for simultaneously and separately detecting both positive and negative ions.

The high frequency switching circuit means preferably comprise a plurality of independently adjustable voltage sources, electronic switching circuit means coupled to the said voltage sources for selectively connecting the said voltage sources with the said repeller, source and lens electrodes, and timing circuit means coupled to the said electronic switching circuit means for controlling the switching frequency thereof.

The apparatus preferably further comprises mode selector switch means coupled to the said timing and electronic switching circuit means for selectively disabling the said timing circuit means and for selectively establishing a fixed condition of the said switching circuit means.

The plurality of voltage sources preferably include at least four independently variable voltage divider means for independently setting desired positive and negative voltage levels.

Output power amplifying means are preferably coupled between the said switching circuit means and the said repeller, source and lens electrodes.

The apparatus in a preferred form of the invention also incorporates dual detector apparatus for simultaneously detector both positive and negative ions comprising a pair of electron multiplier tubes, negative biasing means coupled to one of the said tubes for attracting positive ions and positive biasing means coupled to the said other tube for attracting negative ions. The apparatus may further comprise cross-talk reducing means coupled to the said dual detector means for reducing cross-talk between the said electron multiplier tubes.

The said cross-talk reducing means may comprise a conductive plate including a pair of apertures, the said apertures being positioned to permit ions to pass through the said plate and impinge upon the said electron multiplier tube, and an upstanding separating fin secured to the said plate midway between the said apertures.

The ion generator 20 is conventional in structure and may be operated in either the electron impact or chemical ionization modes. Ion generators of any other type may also be employed (cf. e.g. U.S. Patent 3,555,272 to *Munson et al*, issued Jan. 12, 1971). The ion generator includes a filament electrode 28, a repeller electrode 30, an ion lens 32 and a source chamber 34. In a quadrupole mass spectrometer, all of these elements operate at relatively low voltages, i.e., between roughly five and sixty volts.

A negative/positive ion controller 36 is coupled to the repeller 30, lens 32 and source 34 for the purpose of rapidly changing the potentials of these elements. More particularly, the negative/positive ion controller 36 (referred to simply as "ion controller" hereafter) supplies a square wave having a frequency in the range of 10kHz to the repeller, source and lens. The two levels of the square wave are independently variable in accordance with the circuitry of the present invention.

The typical output voltages of the ion controller 36 are illustrated graphically in Figure 3. The upper square wave curve 38 in Figure 3 represents the voltage applied by the ion controller 36 to both the repeller 30 and the source 34. As shown, this voltage varies between plus and minus five volts. The lens voltage is illustrated by the lower square wave curve 40 and varies between plus and minus ten volts. The illustrated voltage patterns result in the transmission of alternate bundles of positive and negative ions toward the quadrupole filter 15 where they are mass analyzed and subsequently detected. More specifically, positive ions are transmitted when the source and repeller voltages are positive and the lens voltage is negative, while negative ions are transmitted under the opposite voltage polarity conditions.

The details of the ion controller circuit 36 are illustrated in the schematic diagram of Figure 2. As shown in Figure 2 a power supply 42 having outputs of plus five volts, plus and minus fifteen volts and plus and minus sixty volts, as well as a conventional ground is provided to supply required driving power to the illustrated ion controller circuit. The ion controller circuitry includes an isolating amplifier 44, which may be any appropriate dual input amplifier. One input of the amplifier 44 is connected directly to the output thereof, while the other input to the amplifier is connected to the "ion program" output of a conventional quadrupole mass spectrometer, such as the Finnigan device described above. The ion program is a swept DC voltage which is varied in accordance with the mass of the ions being analyzed, allowing the potential of the source 34 to be increased with increasing mass scan.

The output of the isolating amplifier 44 is divided into a positive ion program circuit 46 and a negative ion program circuit 48. The positive ion program circuit 46 includes a coupling resistor 50 connected to the input of a gain control amplifier 52. A variable resistor 54 is coupled in a feedback arrangement across the gain control amplifier 52 to permit adjustment of the gain across the amplifier. The values of the resistors 50 and 54 may be selected so that the output voltage of the amplifier 52 may be varied between zero and one-half of the input voltage. The output of the amplifier 52 is coupled over a line 55 to one input 12 of an integrated switching circuit 56, to be described in more detail subsequently.

The negative ion program circuit includes a gain control amplifier 58, a coupling resistor 60 and a variable resistor 62 coupled across the amplifier 58 to form a circuit that is substantially identical to the positive ion program circuit. An inverter amplifier 64, including coupling and feedback resistors 66 and 68 is, however, coupled to the output of the gain control amplifier 58 in the negative ion program circuit. The output of the inverter amplifier is coupled through a line 70 to a second input 14 of the integrated switching circuit 56.

The switching circuit 56 is preferably a conventional CMOS dual SPOT analog switch conventionally available as a model AD7512 switching circuit from Analog Devices, Inc. The switching circuit includes two output terminals 10 and 13. The input signals received at terminals 9 and 11 are alternately applied to output terminal 10, while the input signals received at terminals 12 and 14 are alternately coupled to the output terminal 13. Of the remaining terminals, terminal 1 is coupled to a source of −15 volts, terminal 2 is grounded, terminals 3 and 4 are coupled to a timing circuit (described subsequently), terminals 5, 6 and 8 are unconnected and terminal 7 is connected to a source of +15 volts. Terminal 9 is coupled over a line 71 to a voltage divider 72, while terminal 11 is similarly coupled over a line 74 to a second voltage divider 76. The voltage dividers 72 and 76 provide offset potentials and permit separate adjustment of the negative and positive source potentials, respectively.

As mentioned previously, the terminals 3 and 4 of integrated switching circuit 56 are coupled together to a point A in a timing circuit 78 illustrated at the lower portion of the figure. The timing circuit includes a conventional integrated circuit timer 80, such as a commercially available Signetics, Inc. model 555 timer. The timer 80 includes the necessary biasing and trimming circuitry, as illustrated at 82 and further includes a variable resistor 84 for adjusting the output frequency. The timer output terminal 3 is connected through a coupling resistor 86 to the point A and to a three-position mode selection switch 88. The three-position switch includes a movable contact 90 which may selectively be coupled to a grounded contact 92, an unconnected or open contact 94 and a contact 96 coupled to the five volt output of power supply 42. The three position switch enables the apparatus of the present invention to be used as both a positive and negative ion generating system (using contact 94), a positive ion system only (contact 96) or a negative ion system only (contact 92). When contact 94 is selected, the timer 80 generates a high frequency output (i.e., in the range of 1−100 kHz) for driving the switching circuit 56 and a second switching circuit described below.

A second integrated switching circuit 98 is provided, and is preferably identical to the switching circuit 56, described previously. The terminals of the integrated switching circuit 98 are connected as follows: terminal 1 to the source of −15 volts, terminal 2 grounded, terminals 3 and 4 to point A, terminal 5, 6 and 8 not connected, terminal 7 to a source of +15 volts and terminals 9, 10 and 11 are interconnected and coupled to ground. Terminal 12 is coupled to a voltage divider 100 for providing the required lens voltage for positive ion detection, while terminal 14 is coupled to a voltage divider 102 for providing the required lens voltage for negative ion detection. Output terminal 13 of switching circuit 98 is connected through a line 104, a coupling resistor 106 and a filtering capacitor 108 to one input of a conventional power output amplifier 110. A suitable amplifier for this purpose is the Burr Brown Model 3581J, a commercially available device. This amplifier includes power input terminals 112 and a trimming potentiometer 114 for balancing the amplifier. As mentioned previously, one input of the amplifier 110 is coupled to the terminal 13 of integrated switching circuit 98. The other input of the amplifier 110 is coupled to a variable resistor 116 connected in a feedback configuration across the amplifier for the purpose of providing gain control. The output of the amplifier is coupled over a line 119 to the lens 32 illustrated in Figure 1.

A substantially identical power output amplifier 118 is connected through coupling resistors 120, 122 and filtering capacitor 124 to output terminal 13 of integrated switching circuit 56. The output terminal 10 of integrated switching circuit 56 is also coupled through coupling resistors 126, 128 and filtering capacitor 130 to the same input of the power amplifier 118. A variable resistor 134 is coupled between the non-grounded input of the amplifier 118 and its output in feedback relationship to provide gain control. As with amplifier 110, appropriate power input leads 136 and a trimming potentiometer 138 are provided with the amplifier 118. The output of the amplifier is supplied over lines 140 and 142 to the repeller 30 and source 34 illustrated in Figure 1.

In operation, the various voltage dividers 72, 76, 100 and 102 are first set to provide the appropriate output voltage levels for the source, repeller and lens voltages, respectively. It should be noted that the voltage levels for generating positive and negative ions are separately adjustable. All other trimming and gain controlling resistors are also set the appropriate values to deliver the proper output gain. All power supply leads are appropriately coupled and the isolating amplifier 44 is coupled to the ion program output of the quadrupole mass spectrometer. The mode switch 88 is then used to select the mode of operation of the device. If the contact 90 engages contact 96, the spectrometer operates in the positive ion mode, and similarly if the contact 90 engages the contact 92, the spectrometer operates strictly in the negative ion mode. In both cases the timer 80 remains inoperative. If the contact 90 engages the contact 94, however, the timer 80 becomes operative and provides triggering inputs to the control terminals 3 and 4 of the integrated switching circuits 56 and 98 for controlling the switching intervals of these circuits. The frequency of the timer 80 is set to an appropriate value in the range mentioned previously for producing two square wave outputs of the type illustrated in Figure 3, one for supplying an appropriate voltage to the source 34 and repeller 30 and a second for applying an appropriate voltage to the lens 32. It is apparent from the previous discussion that the switching circuits operate to alternately apply the signal received on input terminals 12 and 14 to output terminals 13, and similarly to apply the input voltage received at terminals 9 and 11 to output terminals 10 (the latter applies only to switching circuit 56 since switching circuit 98 requires only one output from terminal 13). These output signals are appropriately amplified by the power output amplifiers 110 and 118 to drive the repeller, source and lens of the mass spectrometer. The rapid changes in the potentials of these elements result in the generation of a train of alternate pulses or "bundles" of positive and negative ions. The frequency of the pulse train is, of course, the same as that of the timer 80. If this frequency is 5 kHz, for example, it will be apparent that positive and negative ions reach the detector 26 approximately simultaneously.

Attention is again directed to Figure 1, and particularly to the ion detector 26 illustrated at the right of that figure. As mentioned previously, a single electron multiplier cannot be used to detect both positive and negative ions in view of the fact that electron multipliers are conventionally operated at a high bias potential. Although this bias potential may be either a positive or a negative voltage, whichever voltage is selected tends to repel ions of the same polarity in view of the fact that the ions transmitted through a quadrupole mass spectrometer have very low energy levels. Accordingly, it was necessary to develop a dual multiplier detector apparatus for use with the present invention in order to permit simultaneous detection of both positive and negative ions. The dual electron multiplier apparatus is shown in block diagrammatic form in Figure 1 as including a pair of standard electron multiplier tubes 144 and 146, both of the type conventionally used in mass spectrometers. Galileo continuous dynode multipliers may be used, for example. The outputs of the electron multiplier tubes 144 and 146 are respectively applied to a negative ion preamplifier 148 and a positive ion preamplifier 150. The outputs of these preamplifiers are subsequently fed to suitable conventional data processing equipment, such as an oscilloscope 152, a chart recorder 154 and a computer 156, although other types of analytical equipment may also be used.

The positive ion channel of the electron multiplier system described above is essentially a conventional channel of the type that is standard equipment with the Finnigan mass spectrometer previously referenced. The electron multiplier tube 146 is conventionally biased at −2KV at its input, whereby only positive ions are attracted to it for processing. Any negative ions passing through the quadrupole filter 10 would thus be repelled by the large negative bias on the tube 146, preventing any further detection or processing of negative ions. Accordingly, the negative ion channel added to the apparatus of the present invention is adapted to attract and process negative ions. To do so, the negative electron multiplier tube 144 is biased such that its output is coupled to a voltage source 154 which supplies a bias voltage of approximately +4KV. The input of the electron multiplier 144 is coupled to go ground potential through a large isolating resistor 156, whereby the input of the tube 144 is maintained at a high positive potential for attracting negative ions.

To accommodate the high positive bias of the tube 144, the negative ion preamplifier 148 must be capable of operating at approximately 4KV above ground potential. This requirement is met by conventionally available amplifiers, such as an Extranuclear model 032−4 Negative/Positive Ion Preamplifier.

Figures 4 and 5 illustrate the mechanical structure of the dual electron multiplier structure of the present invention. As shown, the tubes 144 and 146 are secured to a mounting structure or panel 158 preferably formed of a high-grade insulating material such as a conventional high dielectric ceramic material. The mounting structure 158 is secured to a base 160 preferably formed of metal and provided for enabling the dual multiplier structure to be secured to the remaining portions of the mass spectrometer apparatus in a vacuum tight manner.

To prevent cross talk between the positive and negative ion detection channels, an X-ray shield 162 is mounted in front of the input ends of the electron multiplier tubes 144 and 146. The shield 162 is secured to base 160 by means of a metal supporting rod 164 which also serves to maintain the shield 162 at ground potential. The shield 162 includes a disc portion 166 of a diameter sufficient to completely cover the faces of both of the electron-multiplier tubes 144 and 146. A pair of slots 168 and 170 are formed in the disc 166 and are positioned to be adjacent the central input areas of the tubes 144 and 146, respectively. Finally, a dividing fin 172, preferably formed of a conductive material, is secured to the outward face of disc 166 across the diameter of the disc at a point equally spaced from the slots 168 and 170. The dividing fin is of such a height as to very closely approach the electrodes 12 within the quadrupole filter 10 when the mass spectrometer apparatus is fully assembled. When so constructed the dividing fin together with the remaining structure of the shield 162 greatly reduces cross-talk between the positive and negative ion channels.

As mentioned previously, one aspect of the present invention includes operating the apparatus of the invention under appropriate conditions to produce both positive and negative ions. An exemplary set of suitable conditions involves the use of isobutane at one torr as the reagent gas and perfluorokerosene as the internal standard. Electron bombardment of this mixture plus a sample produces $C_4H_9^+$ ion and a population of thermal or near thermal electrons. The $C_4H_9^+$ ions function as a Bronsted acid and protonate most organic samples to form M+1 ions, where M is the molecular weight of the sample. The reagent ion, $C_4H_9^+$, does not react with perfluorokerosene, however, so the positive ion beam consists entirely of $C_4H_9^+$ ions together with sample ions.

In contrast to the above-described situation, the internal standard captures thermal electrons but isobutane and most organic molecules do not. Accordingly, only ions derived from the internal standard, perfluorokerosene, appear in the negative ion output. Since both positive and negative ion spectra are recorded simultaneously in accordance with the principles of the present invention, extrapolation from the known mass of the ions derived from the standard provides an indication of the exact mass of the M+1 ions derived from the sample. The elemental composition of the unknown sample is then easily determined from published tables of compositions and exact masses.

It will, of course, be apparent to those skilled in the art of chemical ionization mass spectrometry that various other reagent gas compositions can also be used to produce the desired output of a variety of different positive and negative sample ions carrying useful structural information in accordance with the teachings of the present invention.

The operation of the present invention will now be described in more detail. A conventional quadrupole mass spectrometer, such as a Finnigan unit of the type previously described is initially modified by the addition of a positive/negative ion controller of the type described in detail above. The positive/negative ion controller is coupled to the repeller, source and lens electrodes of the mass spectrometer for varying the potential of these elements in the manner shown, for example, in Figure 3. The conventional ion detecting system of the mass spectrometer is replaced with the dual multiplier apparatus of the present invention and the mass spectrometer is operated under the preferred conditions set forth above. Once the appropriate controls of the negative/positive ion controller are appropriately set in accordance with the teachings of the present invention, the apparatus is operated normally to provide detection of both positive and negative ions. The use of dual input channel recorders and stereo oscilloscopes facilitates the simultaneous comparison of positive and negative ion data.

Various modifications of the present invention are, of course, possible. The dual electron multiplier housing along with the X-ray shield 162 and dividing fin 172 may be sprayed with graphite, or a suitable equivalent composition, for the purpose of suppressing secondary electron emission. Such a treatment of the system tends to further reduce noise and cross-talk.

Similarly, a Faraday cup system can be used in place of the dual electron multipliers for detecting both positive and negative ions. However, the use of a Faraday cup provides a much lower sensitivity than the electron multiplier system described in detail above.

WHAT WE CLAIM IS:-

1. A method of generating and monitoring positive and negative ions effectively simultaneously using a quadrupole mass spectrometer comprising the steps of:

operating an ion source of said mass spectrometer under conditions favouring the generation of positive and negative ions;

rapidly switching the potentials applied to repeller, source and lens electrodes of the said quadrupole mass spectrometer ion source between selected positive and negative levels; and

separately detecting the said positive and negative ions.

2. A method as claimed in Claim 1, further comprising the steps of:-

producing electrical signals representative of the said positive and negative ions detected in the said step of detecting; and

processing the said electrical signals.

3. A method as claimed in Claim 1 or Claim 2 in which perfluorokerosene is used as an internal standard material.

4. A method as claimed in Claim 3 in which isobutane is used as the reagent gas at a pressure of approximately one torr.

5. A method as claimed in any one of Claims 1 to 4 in which the potentials applied to the said repeller, source and lens electrodes are changed at a frequency in the range between 1 and 100 kHz.

6. A method as claimed in Claim 5 in which the said frequency is approximately 10 kHz.

7. A method as claimed in Claim 1 substantially as specifically described herein with reference to the accompanying drawings.

8. Apparatus for enabling a quadrupole mass spectrometer having an ion source including repeller, source and lens electrodes to effectively simultaneously generate and monitor both positive and negative ions, comprising:

high frequency switching means for alternately applying selected positive and negative potentials to the said repeller, source and lens electrodes; and

dual detector means for simultaneously and separately detecting both positive and negative ions.

9. Apparatus as claimed in Claim 8, wherein said high frequency switching circuit means comprises:

a plurality of independently adjustable voltage sources,

electronic switching circuit means coupled to the said voltage sources for selectively connecting the said voltage sources with the said repeller, sources and lens electrodes; and

timing circuit means coupled to the said electronic switching circuit means for controlling the switching frequency thereof.

10. Apparatus as claimed in Claim 9, further comprising:

mode selector switch means coupled to the said timing and electronic switching circuit means for selectively disabling the said timing circuit means and for selectively establishing a fixed condition of the said switching circuit means.

11. Apparatus as claimed in Claim 9 or Claim 10 in which:

the said plurality of voltage sources include at least four independently variable voltage divider means for independently setting desired positive and negative voltage levels.

12. Apparatus as claimed in Claim 10 or Claim 11 further comprising:

output power amplifying means coupled between said switching circuit means and the said repeller, source and lens electrodes.

13. Apparatus as claimed in Claim 8 substantially as specifically described herein with reference to the accompanying drawings.

14. Apparatus as claimed in any one of Claims 8 to 13 incorporating dual detector apparatus for simultaneously detecting both positive and negative ions comprising:

a pair of electron multiplier tubes,

negative biasing means coupled to one of the said tubes for attracting positive ions; and

positive biasing means coupled to the said other tube for attracting negative ions.

15. Apparatus as claimed in Claim 14, further comprising:

cross-talk reducing means coupled to the said dual detector means for reducing cross-talk between the said electron multiplier tubes.

16. Apparatus as claimed in Claim 15 wherein said cross-talk reducing means comprises:

a conductive plate including a pair of apertures, the said apertures being positioned to permit ions to pass through the said plate and impinge upon the said electron multiplier tubes; and

an upstanding separating fin secured to the said plate midway between the said apertures.

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Agents for the Applicants.

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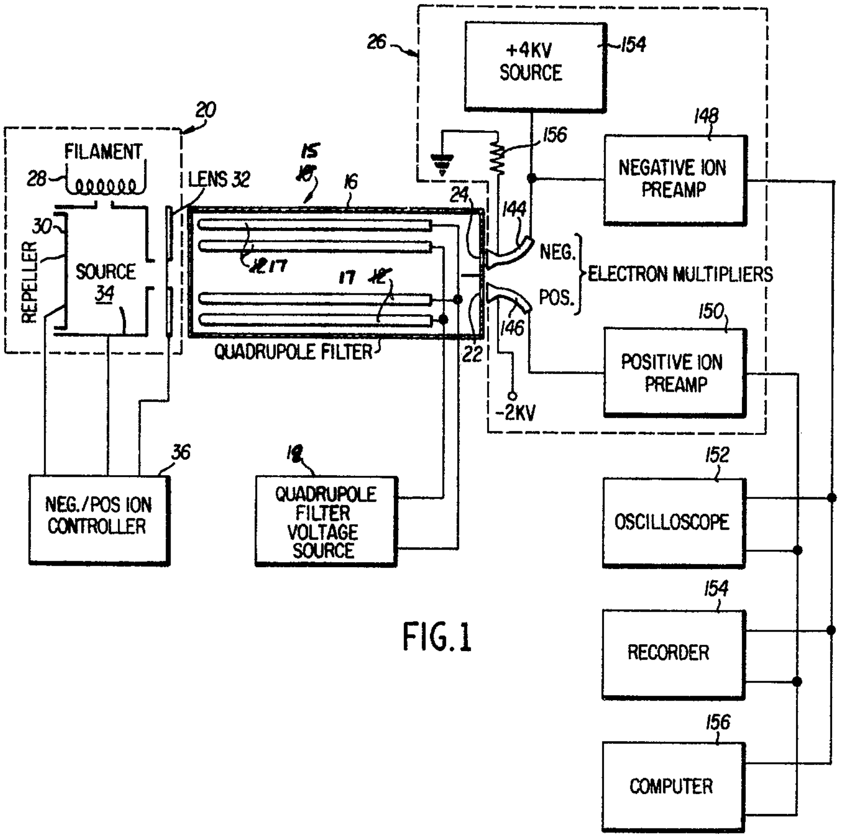
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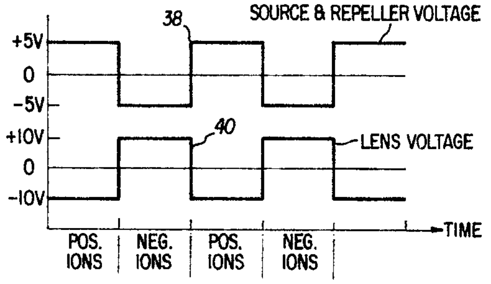
154
148
156
NEGATIVE ION PREAMP
20
FILAMENT
28
15
16
LENS 32
24
144
30
REPELLER
SOURCE 34
17
NEG.
POS.
ELECTRON MULTIPLIERS
146
150
QUADRUPOLE FILTER
22
POSITIVE ION PREAMP
-2KV
36
18
152
NEG./POS ION CONTROLLER
QUADRUPOLE FILTER VOLTAGE SOURCE
OSCILLOSCOPE
154
RECORDER
156
COMPUTER

FIG.1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+5V
0
-5V
+10V
40
LENS VOLTAGE
0
-10V
TIME
POS. IONS
NEG. IONS
POS. IONS
NEG. IONS

FIG.3

1567490 COMPLETE SPECIFICATION

3 SHEETS *This drawing is a reproduction of the Original on a reduced scale*

Sheet 2

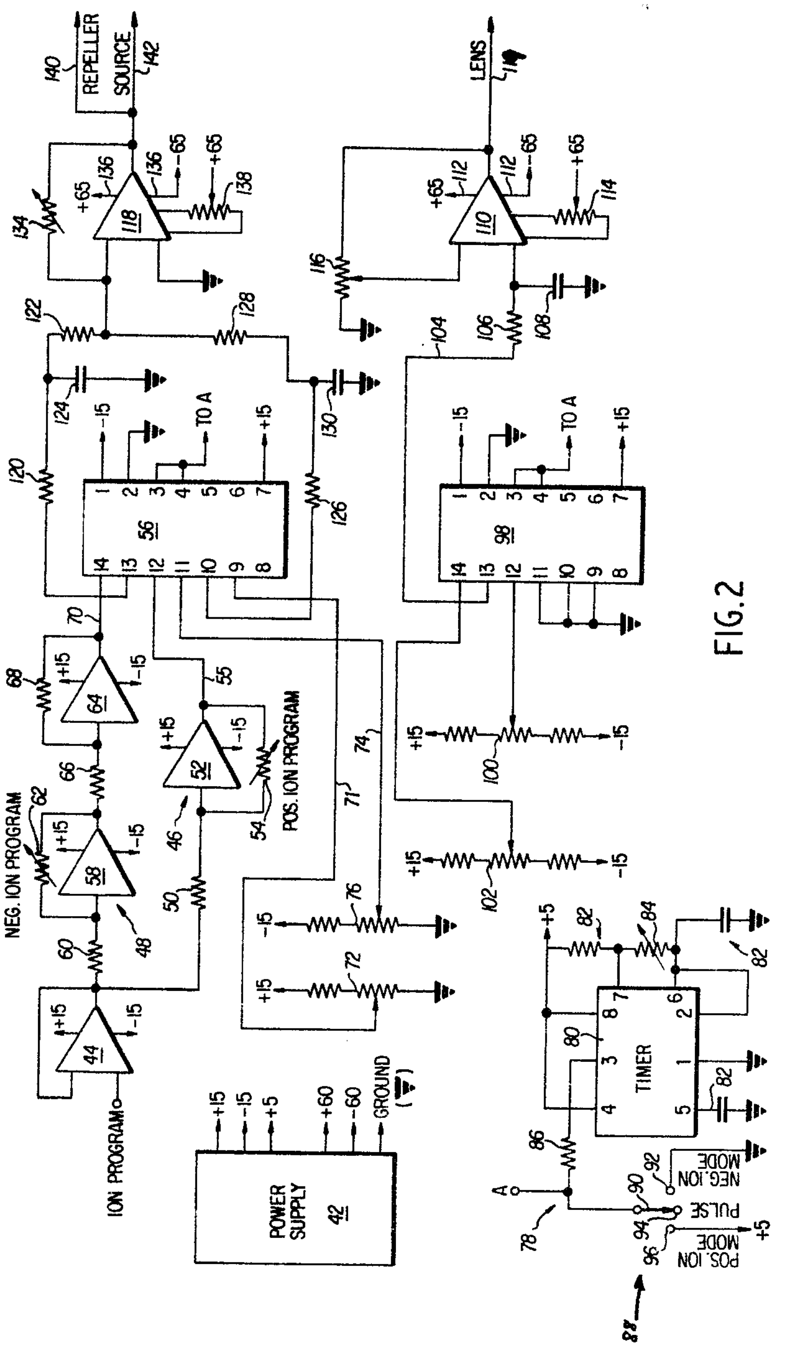
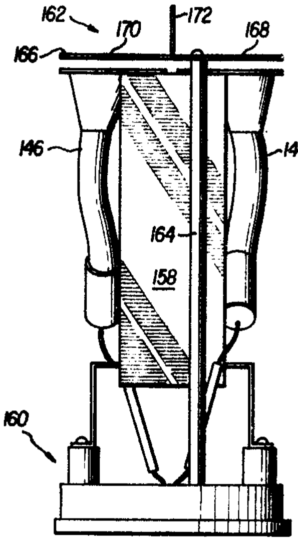


FIG. 2


162
170
172
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146
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FIG.5

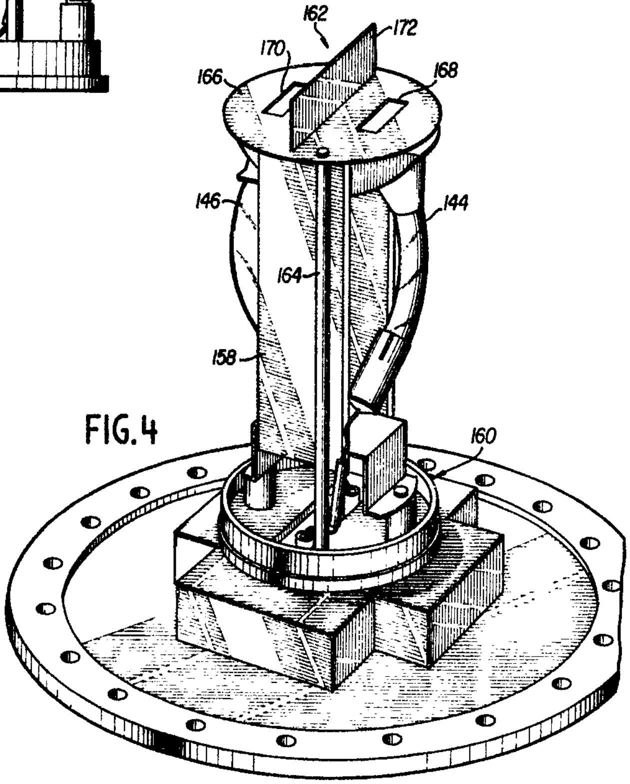
162
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FIG.4