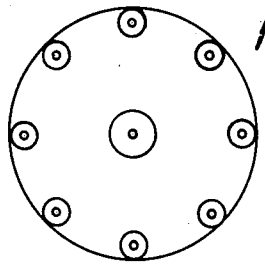
Oct. 4, 1960 W. H. BENNETT 2,955,204

NON-MAGNETIC MASS SPECTROMETER

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FIG. 1



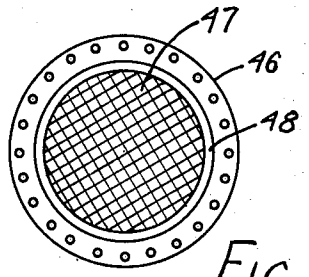
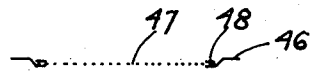
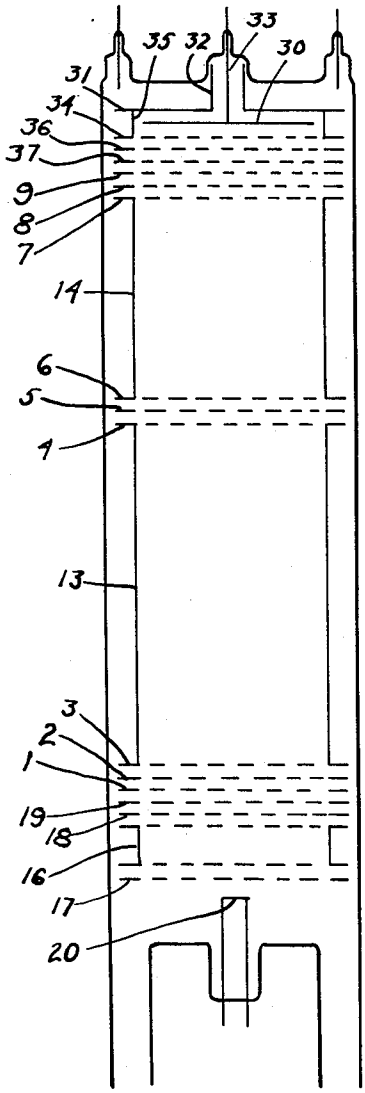


FIG. 3





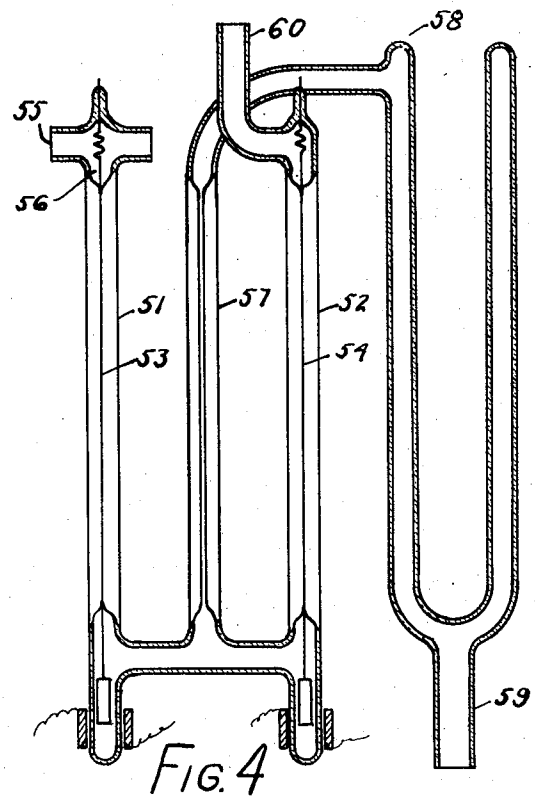


FIG. 4

FIG. 2

INVENTOR

WILLARD H. BENNETT

BY Moore & Hale

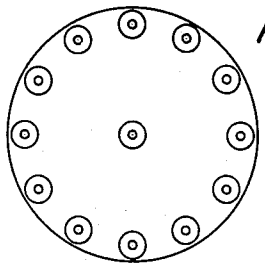
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NON-MAGNETIC MASS SPECTROMETER

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FIG. 5



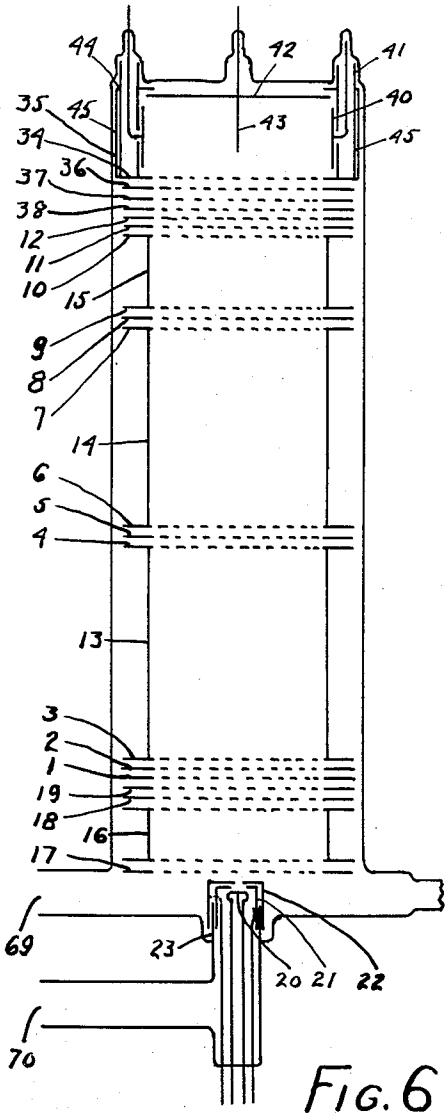


FIG. 6

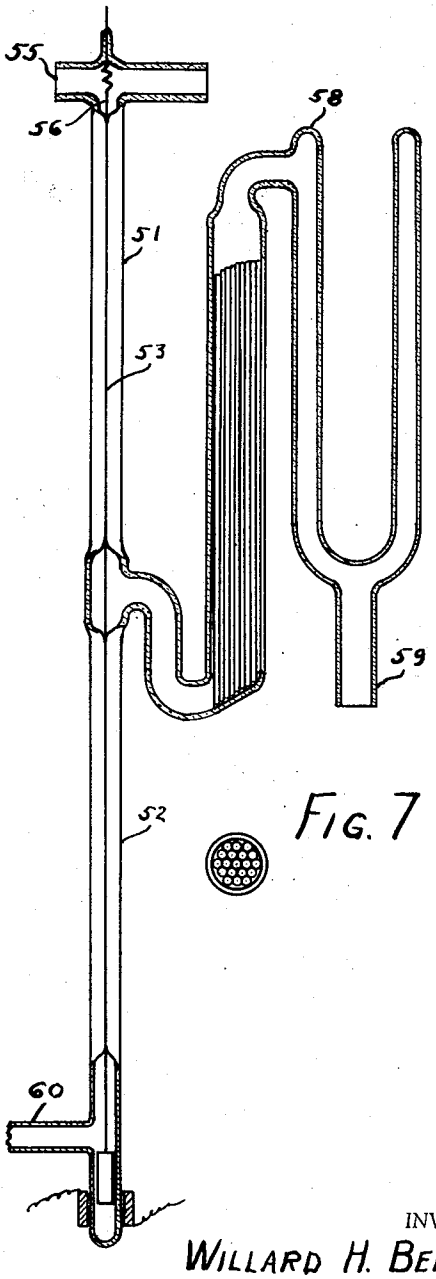


FIG. 7

INVENTOR
WILLARD H. BENNETT

BY Moore & Hall

ATTORNEYS

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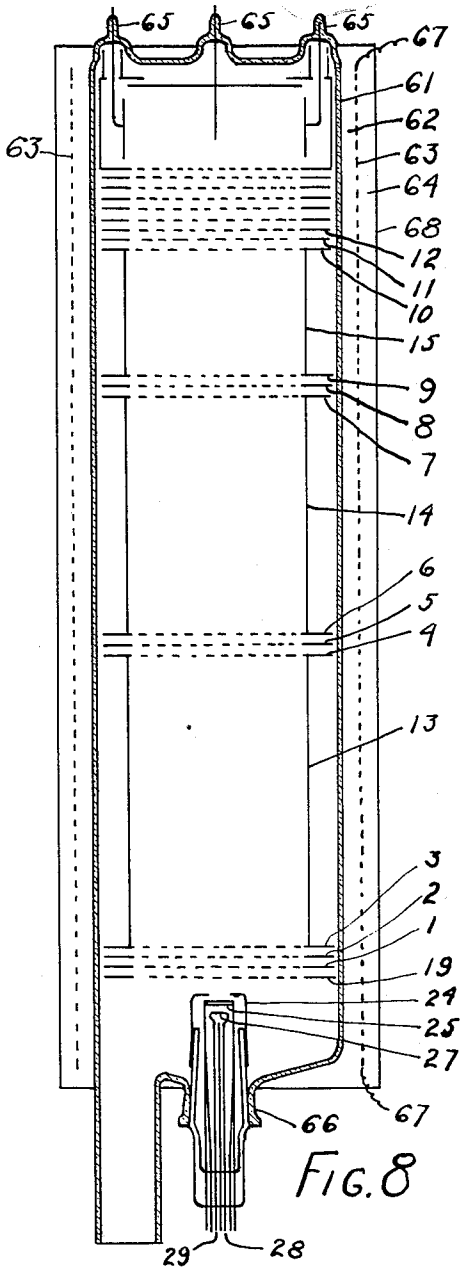


FIG. 8

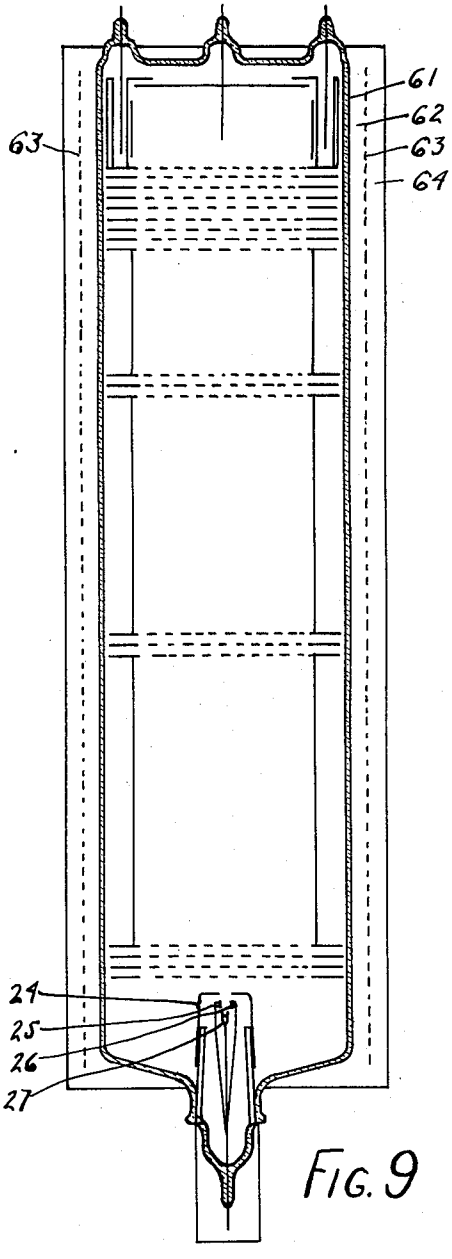


FIG. 9



FIG. 10

FIG. 11

INVENTOR
WILLARD H. BENNETT

BY Moore & Hale

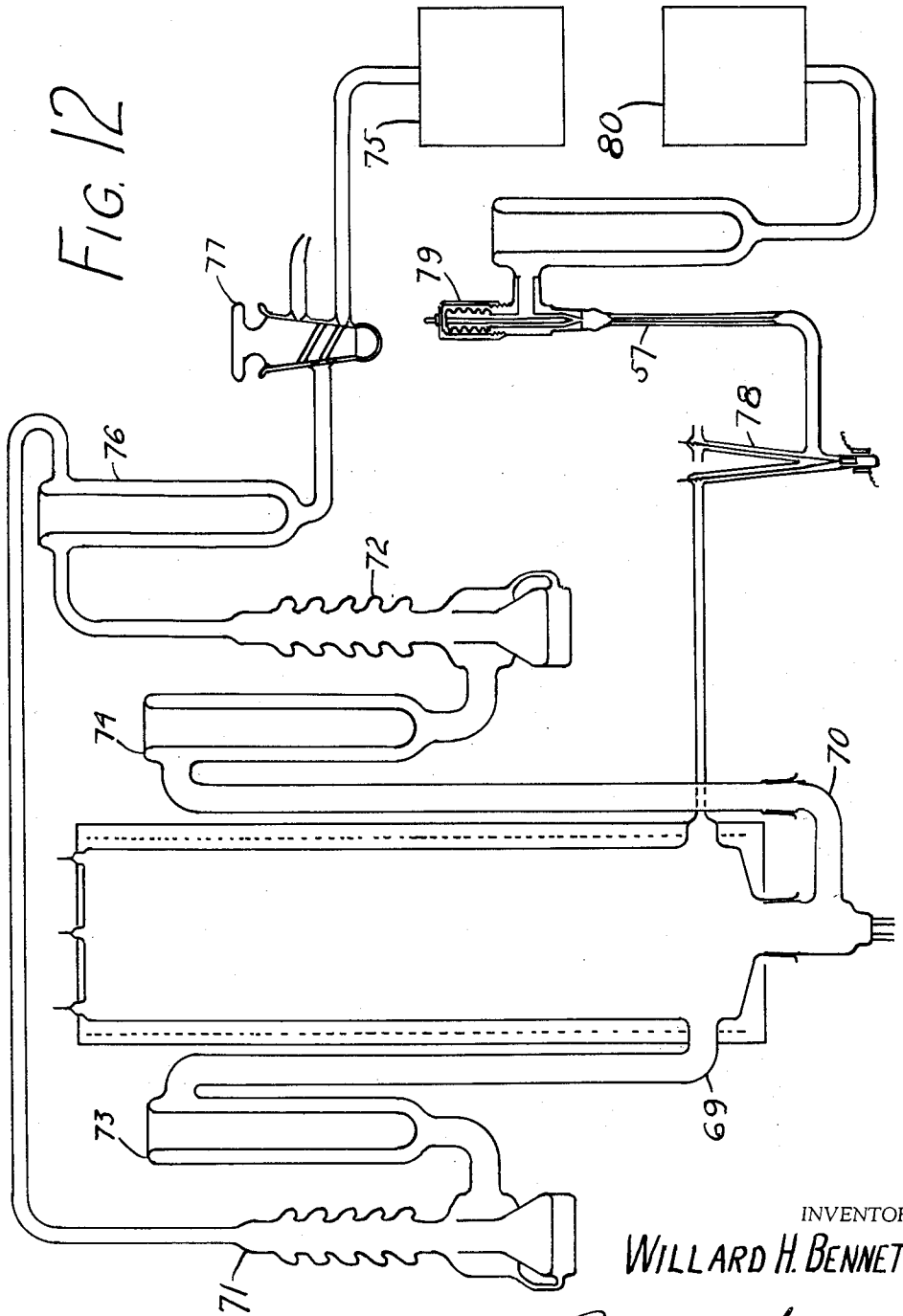
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FIG. 12



INVENTOR
WILLARD H. BENNETT

BY Moore & Hall

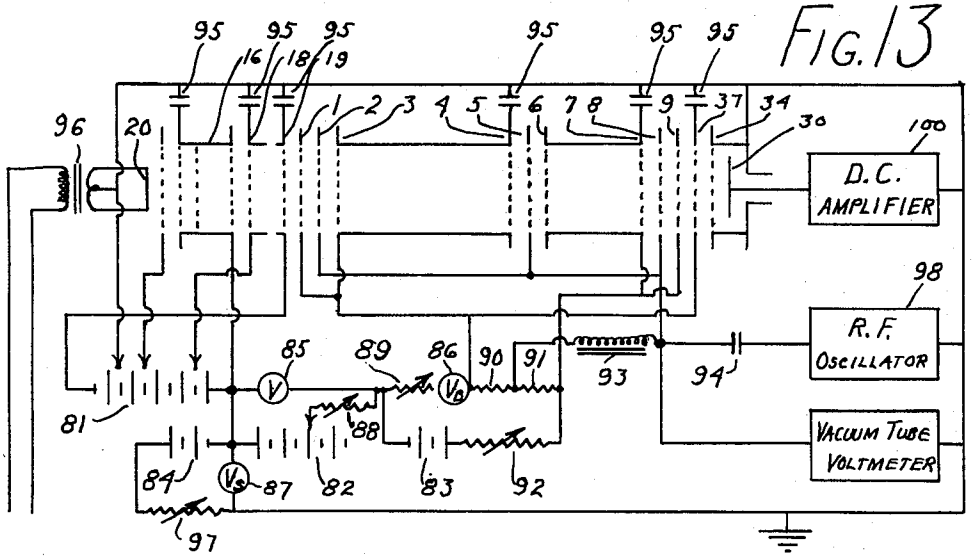
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FIG. 13



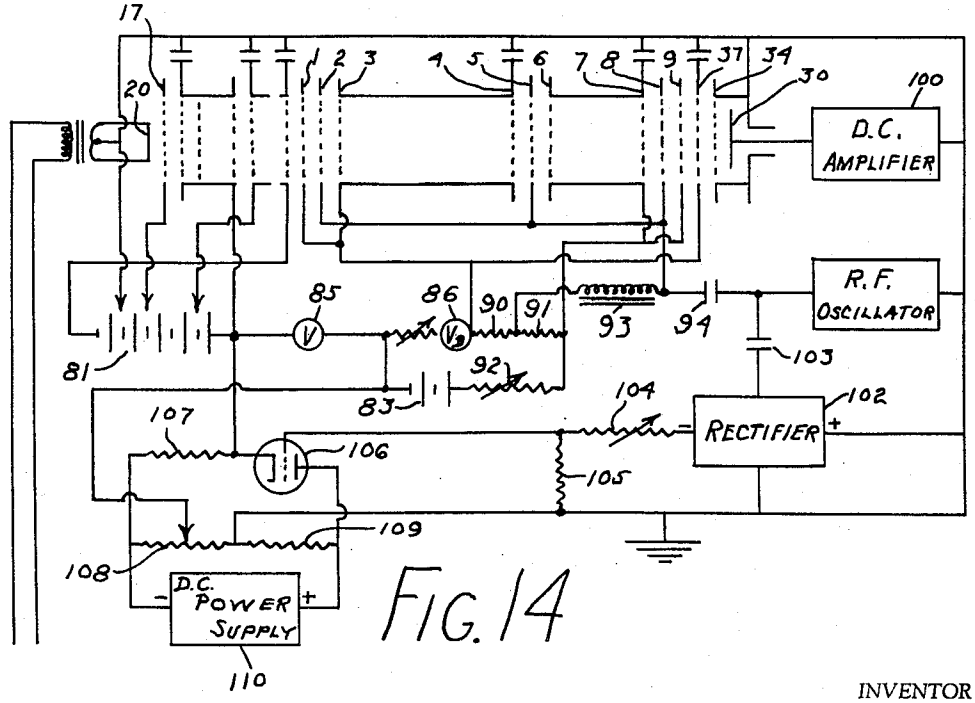


FIG. 14

INVENTOR

WILLARD H. BENNETT

BY Moore & Hall

ATTORNEYS

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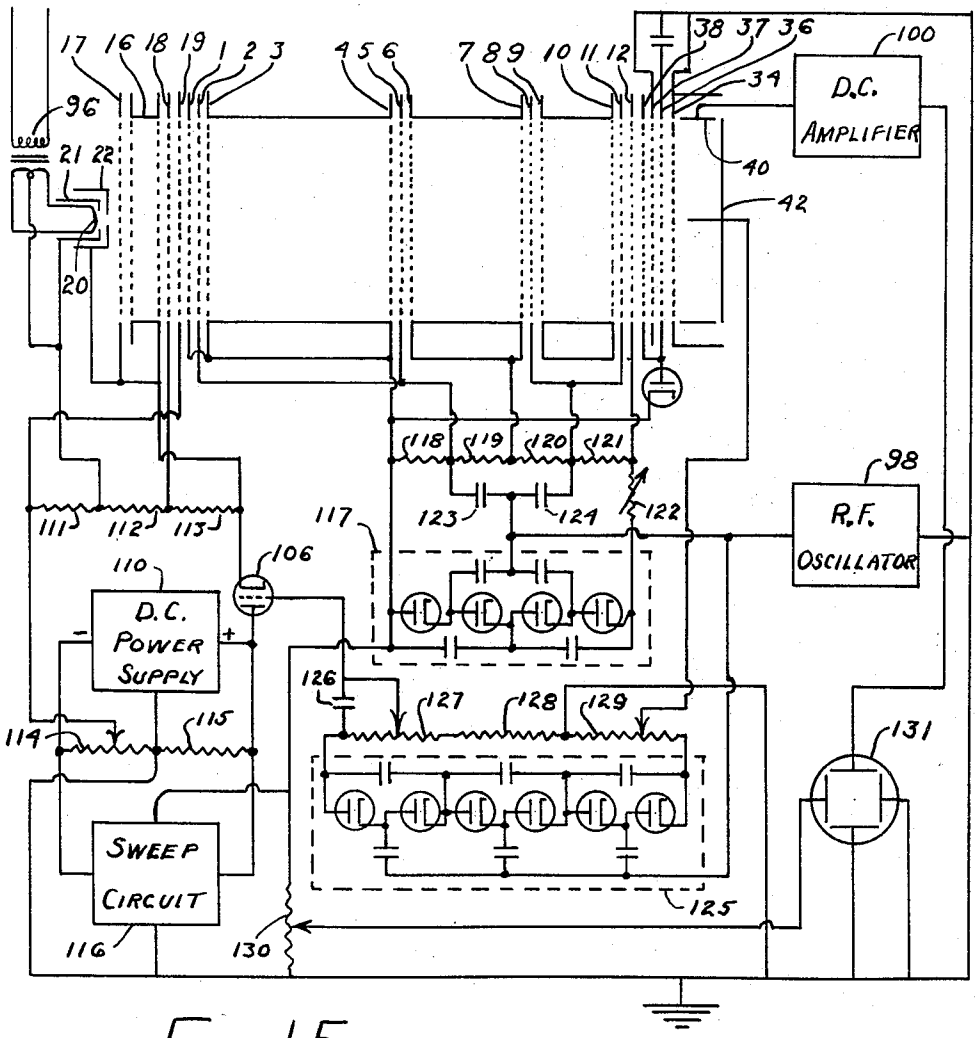


FIG. 15

INVENTOR
WILLARD H. BENNETT

BY Moore & Hall

ATTORNEYS

United States Patent Office

2,955,204

Patented Oct. 4, 1960

2,955,204

NON-MAGNETIC MASS SPECTROMETER

Willard H. Bennett, 4008 1st St. SW., Washington 24, D.C.

Substituted for abandoned application Ser. No. 196,024, Nov. 16, 1950. This application Aug. 30, 1955, Ser. No. 531,337

11 Claims. (Cl. 250—41.9)

This invention relates to mass spectrometers including a velocity selector type of tube and associated apparatus which may be employed in the analysis or separation of ions having different isotopic mass and in the analysis or separation of ions having different chemical identities.

This application is a continuation-in-part of my co-pending application, Radio frequency mass spectrometer, Serial Number 456,983, filed September 20, 1954 (now U.S. Patent No. 2,721,271), which was a substitute for and co-pending with the application of the same name, Serial Number 196,024, filed November 16, 1950, and now abandoned, and this application is also a continuation-in-part of my co-pending application, Leak Primarily for mass spectrometers, Serial Number 241,813, filed August 14, 1951, now U.S. Patent No. 2,721,270.

An object of this invention is to provide a novel tube and associated apparatus wherewith a mixture of substances having different chemical or isotopic identities may be separated into its components and the amount of each such component in said mixture may be measured.

A further object of this invention is the provision of new and novel apparatus employing radio-frequency alternating fields for the separation of ions having different masses or chemical identities.

Still another object of this invention is the provision of a mass spectrometer which is capable of scanning a mass range rapidly enough to yield a continuing presentation of the mass spectrum on a cathode ray oscilloscope screen.

Another object of this invention is the provision of a method and apparatus for accurately determining the isotopic composition of each element present in a mixture.

Still another object of this invention is the provision of a rugged and reliable apparatus for the continuing admission of gases at a controlled rate into a system.

These and other objects may be seen from the following description and correlative drawings in which:

Figure 1 is a top view of a three-stage tube in accordance with my invention;

Figure 2 is a schematic cross-section view of the same tube;

Figure 3 shows the structure of one of the grids used in these tubes;

Figure 4 is a schematic representation of one form of the leak used for admitting gases into a tube;

Figure 5 is an end view of a four-stage tube;

Figure 6 is a schematic cross-section view of a four-stage tube with a cylindrical collector and separately pumped cathode;

Figure 7 is another form of a leak for admitting gases at a controlled rate to the tube;

Figure 8 shows a schematic cross-sectional view of a four-stage tube wrapped with a baking oven;

Figure 9 is a schematic cross-sectional view of the same tube with the section taken at right angles to the section taken in the above;

Figure 10 shows the shape of the opening in front of the cathode in Figure 8;

Figure 11 shows the shape of the cathodes and boat in the ion source of Figure 8;

Figure 12 is a schematic representation of a mass spectrometer tube with its associated vacuum system and leak;

Figure 13 is a schematic representation of the circuitry which may be used with a three-stage tube in accordance with my invention;

Figure 14 is a schematic representation of an alternative form of circuitry which may be used;

Figure 15 is a schematic representation of still another form of circuitry which may be used with a four-stage tube in accordance with my invention.

In a typical embodiment of my invention a sample of gas is introduced at a very low rate of flow into one end of the tube through the medium of a gas leak. It is to be understood that other methods of introducing the material to be analyzed may readily be employed. The gas or vaporized material in the tube is ionized by electrons which are flowing near one end of the tube and the system producing such ions will be known in general throughout the following description as the "ion source." The mass spectrometer tube itself takes the form of an elongated tube; a collector system is placed at the other end of the tube from the ion source and a plurality of grids spaced between the two. To the grids are applied appropriate D.C. and radio-frequency A.C. potentials, these potentials being so selected that the ions of different masses which pass through said grids will acquire varying amounts of energy. By the proper selection of potentials a pre-selected ionic mass will be accelerated more than the other masses present. In the collector system or to the collector plate is applied a potential difference from the ion source which is sufficient to repel all ions approaching it except those of pre-selected mass which alone have the required energy to reach the collector.

A mass spectrometer tube consists of three principal parts: the ion source, the analyzer, and the collector, all of which are enclosed in the tube envelope which is usually of glass but which may be made of other materials.

One form of ion source is shown in Figure 2 wherein electrons from cathode **20** are accelerated towards electrodes **17** and **16** by applying an electrical potential difference such that electrodes **17** and **16** are each positive relative to the potential of the cathode **20**. Electrode **17** is a grid similar to the other grids in the tube which will be described in greater detail later. Electrode **16** consists of two grids and a metallic cylindrical wall shown in cross-section in Figure 2. The two grids and cylindrical wall are mechanically and electrically interconnected. Included in the ion source are also grids **18** and **19** to which electrical potentials are applied such that grid **18** is at a small negative potential difference from cage **16** and grid **19** is at a potential which is negative relative to the cathode **20**. Electrons coming from cathode **20** are accelerated towards cage **16** and most of them pass through the cage ionizing some of the gas which is in the cage. The electrons continue on their way and are turned back by the negative potential on grid **19** many of them making another trip through the cage and ionizing still more of the gas molecules in the cage. In this way the negative potential on grid **19** prevents any of the electrons from the cathode **20** from passing up through the tube beyond grid **19**. The small negative potential on grid **18** relative to the potential of the cage **16** causes the positive ions being produced inside the cage **16** to emerge towards grid **18** and to pass upward into the analyzer to be described in greater detail later.

Another form of ion source is illustrated in Figure 6 in which the cathode **20** is partially covered with an electrode **21** which is shaped in the form of a cylindrical cup with an aperture in the end of the cup. Covering electrode **21** is electrode **22** which is also shaped in the form of a cup with an aperture in the end of the cup. Either electrode **21** or electrode **22** is fitted closely to the cylindrical glass wall **23** so that the only way that the gases in the tube can get to the space around the cathode is by passing through the small apertures in **21** and **22**. In this ion source electrode **22** is held at a potential which is more positive than the cathode but which is not more positive than the potentials on grid **17** or cage **16**. The potential on electrode **22** draws electrons away from the cathode **20** while the potential on electrode **21** is held at a value which is equal to or only a little different from the potential on the cathode. The potential on electrode **21** may be either a little positive or a little negative or equal to the potential on the cathode **20**. In this way the potential applied to electrode **21** tends to guide the electrons through the aperture in electrode **22** after which the electron stream spreads out somewhat while passing through grid **17** and cage **16**. The cage **16** and the grids **17, 18** and **19** serve the same purposes and are operated in the same ways as before.

Still another form of ion source is illustrated in Figures 8 and 9 which is an ion source for the analysis of solid materials. The sample of material to be analyzed is placed in a boat-shaped ribbon electrode **27**. Located above the boat are either one or two parallel cathodes **25** and **26**. Covering the cathodes and the boat there may be an electrode **24** shaped in the form of a cup in end of which is a rectangular-shaped aperture like that shown in Figure 10. In Figure 11 are shown the top view of the boat **27** and two parallel cathodes **25** and **26**. In this ion source electrons from the cathodes **25** and **26** are accelerated towards the boat **27** which is held at a potential which is positive relative to the cathodes. The electron current bombarding the boat heats up the boat. If this heating is insufficient to vaporize the solid sample material in the boat an electrical heating current can be passed through leads **28** and **29** to further heat the materials. The electrode **24** is held at a potential which is negative relative to the cathodes and the potential on grid **19** is also negative relative to the cathodes. The vaporization of the material in the boat produces a vapor through which the electrons from the cathodes must pass and in so passing some of the vapor molecules are ionized. The positive ions so produced are accelerated towards the negative electrodes, that is to say they are accelerated through the space between the two cathodes **25** and **26**, then through the rectangular aperture in electrode **24** and then through the negatively charged grid **19** and so into the analyzer.

The analyzer in a mass spectrometer tube consists of three or more groups of grids each group consisting of three grids and the space between groups of grids surrounded by cylindrically shaped electrically conducting shields.

One form of analyzer is illustrated in Figure 2 in which grids **1, 2** and **3** comprise the first group or stage, grids **4, 5** and **6** comprise the second stage, and grids **7, 8** and **9** comprise the third stage of this three-stage tube. Mechanically and electrically connected to grids **3** and **4** is the electrical shield **13** and similarly connected to grids **6** and **7** is the shield **14**. It is convenient but not necessary to electrically connect grid **1** to electrodes **3, 13** and **4** and similarly to connect grid **9** to electrodes **6, 14** and **7**. It is also convenient but not necessary to interconnect electrodes **2, 5** and **8**. The distance from grid **2** to **5** divided by the distance from grid **5** to grid **8** equals the ratio of two integral numbers. These are preferably two prime numbers and good numbers to use for this three-stage tube shown in Figure 2 are 7 and 5. That is to say the distance from grid **2** to grid **5** divided by the distance from grid **5** to grid **8** equals exactly 7 divided by 5. Other integers may be chosen in this ratio but in any event they must be different integers else it will be found to be impractical to obtain good sensitivity in the separation of ions having different masses. The distance from grid **2** to grid **5** divided by the corresponding integer (which is 7 in the example given above) is equal to the distance from grid **5** to grid **8** divided by its corresponding integer (which is 5 in the example given above) and these two equal quotients will be called the interval and is equal to the distance an ion travels during a period of the radio frequency potential for that species of ion having the particular mass which permits it to acquire enough energy from the radio frequency fields to reach the collector as will be explained later. The distance between the three grids in each stage is equal approximately to three eighths of the interval. In the example given above the distance from grid **1** to grid **2** equals the distance from grid **2** to grid **3** equals the distance from grid **4** to grid **5** equals the distance from grid **5** to grid **6** equals the distance from grid **7** to grid **8** equals the distance from grid **8** to grid **9** and equals three fifty-sixths of the distance from grid **2** to grid **5** and equals three fortieths the distance from grid **5** to grid **8**. In measuring these distances between grids these various distances are all measured to the mid-plane of a grid and not to the nearer side of a grid and more particularly the distances are measured to the mid-plane of the knitted portion of the grid rather than any part of the supporting structure of that grid. For example if the knitted portion of the grid has a thickness of 0.003" and it is desired to make the distance between grid **1** and grid **2** equal to 0.121" then the distance between the nearer portions of the knitted portions must be made equal to 0.118".

One form of analyzer for a four-stage tube is illustrated in Figure 6. In this analyzer grids **3** and **4** are mechanically and electrically interconnected with the shield **13**, grids **6** and **7** are interconnected with shield **14** and grids **9** and **10** are interconnected with shield **15**. It is desirable but not necessary for grid **1** to be electrically connected to **3, 13, 4** and for grid **12** to be electrically connected to **9, 15, 10**. Grids **2** and **5** are electrically interconnected and are connected to one of the lead-outs for the application of radio frequency potential (in addition to D.C. base potential) and grids **8** and **11** are interconnected and are connected to another lead-out for a similar application of radio frequency and D.C. potential. Here again in the four-stage tube the distance from grid **2** to grid **5** divided by an integer equals the distance from grid **5** to grid **8** divided by a different integer and this equals the distance from grid **8** to grid **11** divided by an integer which is different from either of the other two integers, and these three equal quotients are equal to the interval three-eighths of which equals the distance between any two adjacent grids in each stage. A good choice of integers for a four-stage tube is 13, 11 and 7 and another good choice of integers is 17, 13 and 11. Other choices of integer combinations may be used with good results and not all the integers need be prime numbers.

One form of collector system is illustrated in Figure 2 wherein a collector plate **30** is surrounded by an electrical shield consisting of a backing plate **31** which has a small cylindrical sleeve **32** surrounding the supporting post **33**, a grid **34** and a cylindrical shield **35** where **31, 32, 34**, and **35** are electrically and mechanically interconnected, and grids **36** and **37**. Either or both of grids **36** and **37** may be replaced with a pair of grids electrically and mechanically interconnected for improved operation and where such grid doublet is used a reference to grid **36** or grid **37** respectively will be meant to mean such a doublet.

An alternative form of collector system is illustrated in Figure 6. In this collector the cylindrical electrode **40** constitutes the collector electrode and is not electrically

connected to any other part of the tube but is provided with its own separate and shielded lead-out. One of the metallic supports of this cylinder may be used as the lead-out and that lead-out is surrounded by and insulated from a shielding sleeve 41. The plate 42 looks much like the collector plate 30 of the previous form of collector but the plate 42 serves a different purpose. This electrode 42 has attached to its center a post 43. A shielding sleeve 35, a backing annular ring 44 and grid 34 are electrically and mechanically interconnected and extending from grid 34 to ring 44 are a number of small cylindrical shields 45, 45 one for each of the leads extending from the head of the tube to the respective electrodes below. The purpose of these shielding sleeves is to prevent any electrical fields due to fluctuations in the electrical potential on any of these leads from reaching the collector cylinder 40. Grids 36 and 37 and a grid 38 are also a part of this collector system.

In the construction of these tubes a convenient material to use is knitted fine wire mounted in a stretched condition on metal rings as illustrated in Figure 3 in which a plan-view and cross-sectional view are shown. In this figure the knitted wire is shown with rectangular crossed lines at 47 but in actual fact the knitted wire consists of interlocking knitted loops which are more or less curved everywhere in a manner which is quite similar to the structure in a knitted nylon stocking. This material is stretched over a narrow annular metal ring 48 lying on a stretching form for the knitted wire and a formed grid ring 46 which has been punched with a number of holes as shown, is laid over the knitted material and welded fast by electric welding to the ring 48. This welding of ring 46 to 48 is done at many places as closely spaced as possible all around the rings so that the knitted material is crushed and held firmly between the welded rings. There is of course some electric welding of the wire to either or both rings since the wire lies between the two rings. After such welding the excess knitted material lying outside ring 48 is trimmed away. A suitable knitted material is one mil tungsten wire knitted with about 16 loops per inch. Another suitable material is ½ mil tungsten wire knitted with about 25 loops per inch.

In the fabrication of these various forms of mass spectrometer tubes, there are many different methods for supporting the electrodes inside the envelope and for making electrical connections through the wall of the envelope to the electrodes, which will be evident to anyone skilled in the vacuum tube art.

In the operation of this mass spectrometer an ion which passes the first grid of any three-grid stage while the middle grid has a negative charge on it momentarily due to the radio frequency potential being applied to that middle grid, and which passes through the middle grid at approximately the time when the charge on that middle grid passes through zero so that the charge on that middle grid becomes positive while the ion is passing from the second to the third grid, the ion is said to be resonant with that stage. Such a resonant ion is accelerated by the radio frequency field both before and after passing through the middle grid and by virtue of such acceleration a resonant ion acquires energy in addition to the energy that it would have had by virtue of the steady potentials applied to the grids. For example, suppose that potentials are to be applied in the spectrometer represented in Figure 2 to make an ion travel through each successive drift space with 100 electron-volts of kinetic energy. In this case it will be said that the principal D.C. potential V is 100 volts. Suppose also that the radio frequency potential being applied to grids 2, 5 and 8 is 5 volts root mean square, in which case the potential difference to apply between the cage and the electrodes 1, 3 and 4 is 90 volts. An ion produced in the cage 16 emerges through grids 18 and 19 to arrive at grid 1 with a kinetic energy equal to 90 electron-volts, if the ion is singly charged. An ion which can be resonant in all three stages of this tube would have to pass each of grids 2, 5 and 8 just when the charge on each of those grids is changing from negative to positive and is passing through zero. Such resonant ion in passing from grids 1 to 3 acquires an additional kinetic energy of approximately 10 electron-volts (that is about twice the root mean square value of the radio frequency potential.) Thus the ion enters the drift space between grids 3 and 4 with a kinetic energy of 100 electron-volts. There is a bias potential applied between drift space 3, 13, 4 and drift space 6, 14, 7 equal to 10 volts so that if no radio frequency potential were applied to grid 5 the ion from the first drift space would enter at second drift space with its kinetic energy reduced to 90 electron-volts. Such an ion would also have 10 electron-volts of potential energy so that its total energy is still 100 electron-volts. However, because the radio frequency potential is applied to grid 5, the resonant ion passing through the second stage acquires an additional kinetic energy of 10 electron-volts so that that ion enters the second drift space with a kinetic energy of 100-electron-volts in addition to its 10 volts of potential energy. This resonant ion in passing through the third stage acquires an additional kinetic energy of 10 electron-volts so that this ion emerges through grid 9 with a kinetic energy of 110 electron-volts in addition to its 10 electron-volts of potential energy and the total energy of the ion is 30 electron-volts greater than when it passed through grid 1 and entered the analyzer. If the collector plate 30 is held at the same potential as the ion source such a resonant ion will reach the collector plate and strike it with a kinetic energy equal to that 30 electron-volts of energy which the ion acquired from the three stages. If the collector plate 30 is held at a potential of approximately 25 volts positive relative to the ion source, the resonant ions can still reach the collector plate striking it with a kinetic energy of about 5 electron-volts. Ions having other masses than the mass which an ion must have in order to be resonant acquire less energy than 30 electron-volts and in fact acquire less than 25 electron-volts of energy from the three stages so that the only kind of ions that can reach the collector are the ions having just the mass-to-charge ratio of the resonant ions. If the tube is a 7 and 5 cycle tube the resonant ions have just the right mass-to-charge ratio to have just the right velocity so that they travel from grid 2 to 5 while the radio frequency potential executes 7 cycles and from grids 5 to 8 while the radio frequency potential executes 5 cycles. The above has been given as an illustrative example only and the tube can be operated in other ways. As one such example instead of applying the retarding potential of 25 volts on the collector 30, a retarding potential could be applied to one or more of the intervening grids 37, 36, or 34 while some of the other grids and possibly the collector plate 30 are held at a negative potential for the purpose of suppressing secondary electron emission. In the appended claims the term energy will be used to designate total energy including both kinetic and potential energies.

As another illustrative example of the operation of a spectrometer tube the structure in Figure 6 may be considered. Let it be supposed that this is a 13, 11 and 7 cycle tube. Here again for an ion to be a resonant ion the ion must pass each of grids 2, 5, 8 and 11 just as the charge on that grid is changing from negative to positive and that ion must pass from grid 2 to 5 while the radio frequency potential on each executes exactly 13 cycles, must pass from grid 5 to 8 while the radio frequency potential on each executes 11 cycles and must pass from grid 8 to 11 while 7 cycles are executed. An ion passing from the ion source into the drift space 3, 13, 4 loses potential energy equal to the charge on the ion times the potential difference between the ion source and the drift space and gains kinetic energy equal to that loss in potential energy plus the energy gained from the first stage

namely twice the root mean square value of the radio frequency potential applied to grid 2. In like manner the resonant ion gains kinetic energy from each of the other stages. One way of connecting the electrodes in the collector system is to interconnect grid 38 with 1, 13, 4 and because of the bias potentials applied between successive drift spaces, grid 38 is negative with respect to grid 12 by four times the root mean square value of the radio frequency potential. Grid 37 can be connected to the retarding potential which in this four-stage tube may be equal to approximately 6½ times the root mean square value of the radio frequency potential such that grid 37 is positive relative to the ion source. Grid 36 may be held at a potential equal to that applied to grid 38 or if rapid scanning of mass range is being performed grid 36 may be held at the maximum negative potential which is applied to 38. Grid 34 and its interconnecting electrodes may be held at a steady potential approximately equal to that of the ion source. Plate 42 with its associated plate 43 is held at 8 times the root mean square value of the radio frequency potential, positive relative to the ion source so that resonant ions from the ion source cannot quite reach that plate 42. The collector 40 is held at the retarding potential which may be about 6½ times the root mean square of the radio frequency, positive relative to the ion source. In this way, the resonant ions are deflected by the potential on electrode 42 and go to the collector 40.

The gases or vapors to be analyzed must be admitted to the mass spectrometer tube at a slow enough rate in order that the pumps which are evacuating the mass spectrometer tube can keep the gases or vapors to be analyzed at a low enough density in the mass spectrometer tube for the mean free path of the ions produced in the ion source to be long compared with the length of the mass spectrometer tube. The pressure of the gases or vapors in the mass spectrometer tube should in most instances be three hundredths of a micron of pressure or less. One method of admitting gases or vapors at such a slow rate is illustrated in Figure 4. Two thick walled capillary glass tubes 51 and 52 have in them wires 53 and 54 respectively. A convenient size is capillary tubes each five centimeters long with bores equal to 0.50 millimeter containing platinum or tantalum wires each 0.48 millimeter in diameter. It is convenient but not necessary to spring-mount each wire at the upper end and to attach a piece of soft iron at the lower end. If a piece of soft iron is attached at the lower end of the wires in the fine capillaries, a coil of wire may be placed around the tube and a little below the level of the piece of iron. Passing a current through the coil draws the iron down and loosens any obstructions which may have collected between the wire and the fine capillary wall. The gas or vapor is admitted at the opening 55 or if the material to be analyzed is a liquid whose vapor is to be used the liquid may be laid in the recess 56. Below the lower ends of the two capillaries a closed glass connection is made with another thick-walled capillary tube 57 convenient dimensions for which would be length 5 centimeters and 1 millimeter bore. It is convenient but not necessary to make connection from the upper end of the large bore capillary and the mechanical pump through a trap 58 the lower end of which 59 is connected to a mechanical pump. The upper end 60 of capillary tube 52 is connected to the mass spectrometer tube. An alternative form of this kind of leak is shown in Figure 7 in which the same wire passes down through both fine capillaries. In place of the coarse capillary may be used a number of small bore thin-walled glass tubes filling the cross-section of a large diameter tube.

The above-stated dimensions of the capillary tubes 51 and 54 together with those of wires 53 and 54 result in viscous flow of the gases in the tubes. Whether the flow is molecular or viscous depends mainly on the relative length and cross-section of the opening rather than the gas flowing therethrough. This invention contemplates that, so far as the claims relating to the leak of Figures 4 and 7 are concerned, the space between the wires 53 and 54 and the inside of the tubes 51 and 52 is much longer than the transverse dimension. The result is substantially viscous flow.

It is convenient to wrap the mass spectrometer tube with a baking oven as shown in Figures 8 and 9. In this oven a thermal insulating material such as asbestos, fibreglass, or any one of the equivalent materials is applied over the glass envelope of the mass spectrometer tube 61. This application of insulating material is shown in Figures 8 and 9 at 62. A coil of heating wire 63 is wrapped over the layer of insulating material 62 after which more heat insulating material is applied over the heating coil at 64. Thermal insulation is applied at both ends of the tube taking care not to cover up the lead-outs such as 65, 65 and 65 or any tapered glass joints such as 66 which may be used as waxed joints and which must be kept exposed to the air in order to keep them cooled to below the softening point of the wax while the rest of the mass spectrometer tube is being baked. The lead-outs from the heating coil are shown at 67, 67. The entire baking structure may be enclosed in a thin metal case 68.

It will be recalled that one form of electron source used in the ion source is illustrated in Figure 6. This tube is evacuated through pumping arm 69 while the space around the filament which serves as the electron source is evacuated by a separate diffusion pump through pumping arm 70. One convenient form of the associated leak and pump systems is illustrated in Figure 12. The mass spectrometer tube is evacuated through arm 69 by a diffusion pump 71 while the space around the filament in the electron source is evacuated through arm 70 by diffusion pump 72. Associated with the diffusion pumps are traps 73 and 74 which are optional if oil diffusion pumps are used but which are necessary if mercury diffusion pumps are used. Both diffusion pumps can be backed by the same backing pump 75 and interposed may be an additional vapor trap 76 and two-way stopcock 77. The gases or vapors to be analyzed are admitted from a leak 78 in which a needle valve 79 may be interposed between the coarse capillary 57 and the mechanical pump 80. A further simplification is that the two pumps 75 and 80 may be driven by the same motor.

Various methods may be used for applying the proper potentials to the electrodes in the mass spectrometer described in this invention as may be seen from the following. One kind of circuit for use with a three-stage mass spectrometer tube is shown in Figure 13. In this form of the invention the filament 20 which serves as the electron source is shown heated from the transformer 96 but a battery could just as well be used for this purpose. A potential difference between the filament 20 and the cage 16 is obtained from a battery 81. The electron current passing from the filament to the cage can be controlled and held steady by use of a grid 17 whose potential difference from the filament is obtained from the same battery 81. The drawing-out potential applied to grid 18 is obtained from the same battery 81 and the potential on grid 19 which is negative relative to the filament is also obtained from the battery 81 as shown. The positive end of battery 81 which is connected to the cage is also connected to the positive end of battery 82 from which the principal D.C. voltage V is obtained and measured on the meter 85. A variable resistor 88 may be connected as shown for the purpose of varying the value of V. The bias potential applied between the successive drift spaces and the D.C. base potential for the radio frequency grids is obtained from battery 83 connected with its negative end toward the negative end of the meter 85. The resistance of each of the resistors 90 and 91 is selected to be equal to ½ the resistance of the bias meter 86. The potential drop across 86, 90, and 91 is adjusted with the variable resistor 92 until the voltage read by the bias meter 86 equals just twice the root mean square value of the radio frequency

potential output of the oscillator **98** as read by the volt meter **99**. The retarding potential applied between the cage **16** and the collector 30 is obtained from the battery **84** in series with which is the variable resistor **97**. The retarding potential so applied is measured by the stopping potential meter **87** and is usually between 4 and 6 times the root mean square value of the radio frequency potential. Interposed between the oscillator **98** and the radio frequency grids **2**, **5**, and **8** is a condenser **94** and interposed between those radio frequency grids and the point between resistors **90** and **91** is a radio frequency choke **93**. Any or all of the condensers **95**, **95**, **95**, **95**, **95**, **95**, may be connected between the respective steady potential electrodes and ground for the purpose of by-passing the capacitive leakage of radio frequency potential which otherwise unintentionally might appear on those electrodes.

An alternative form of circuit is illustrated in Figure 14. In this circuit the output of the RF oscillator **98** is passed through condenser **103** into the cascaded rectifier **102** the positive end of which is grounded. The negative end of the output of the rectifier is passed through a variable resistance **104** and the fixed resistance **105** the values of which are so selected that the potential of the point between them is approximately 5 times the root mean square value of the output of the oscillator **98**. The point between the resistors at that potential is connected to the grid of the cathode follower tube **106** which is driven from a D.C. power supply **110** which is connected as shown to resistors **107**, **108** and **109**. Connected in this way the D.C. power supply **110** also provides the principal D.C. voltage V as measured by volt meter **85** and selected by the variable contact on the resistor **108**. The other connections are similar to the connections used in the previous figure.

Still another kind of circuit which can be used is illustrated in Figure 15 in which a four-stage tube is shown operated entirely by electronic means. In this figure the D.C. power supply **110** supplies the potential to drive the cathode follower tube **106** whose cathode resistance is made up of the three resistors in series **111**, **112** and **113** the points between which are used also for applying the appropriate potentials to the various electrodes in the ion source as shown. The center grounded D.C. power supply **110** is also used to supply the potential for the sweep circuit **116** which may be either a sawtooth or a zigzag sweep circuit. The output of the RF oscillator **98** is used to drive each of the two cascaded rectifiers **117** and **125**, and it is also used for supplying radio frequency potential through the condensers **123** and **124** to the radio frequency grids as shown. The output of the cascaded rectifier **117** supplies potential to the four equal resistors in series **118**, **119**, **120** and **121** which are in series with the variable resistance **122**. The resistance **122** is adjusted until the potential drop across each of the four fixed resistors is equal to the bias potential which as explained before is equal to the root mean square value of the radio frequency potential from oscillator **98**. The cascaded rectifier **125** supplies potential to the three resistors in series **127**, **128** and **129**. The total resistance of **127** is about equal to the total resistance of **129** and the resistance of **128** is about equal to three times either of these. The variable contact on **129** is adjusted to give complete cut-off of resonant ions reaching electrode **42** and the variable contact on **127** is adjusted to give the correct retarding potential in the collector system as explained previously. A part of the sweep potential is taken off from potentiometer and applied to the horizontal plate pair of oscilloscope **131** and the output of the D.C. amplifier **100** is applied to the vertical plate pair in the oscilloscope.

The above circuits are intended to be illustrative only and many other forms of circuit and combinations of components of such circuits appropriately devised to operate mass spectrometer tubes of three or more stages will be evident to anyone skilled in the art. For example instead of the D.C. amplifier with a meter or instead of the cathode ray oscilloscope there may be substituted any one of a variety of kinds of recorders, with appropriate connections to the control means for the principal D.C. voltage V.

Because of the large efficiency with which this kind of mass spectrometer utilizes the output of the ion source it is practical to use large voltage differences between the cathode and the cage, say in the order of 100 to 1000 volts instead of the more usual 50 to 100 volts with the result that there is an enhanced relative yield of multiply charged ions. This makes possible the use of stable isotopes for tracer investigations. For example tracer investigations can be performed in biological and physiological research using materials containing enriched stable isotopes of such elements as carbon, nitrogen, oxygen and others. By comparing the intensities of mass lines appearing at positions 6 and 6½ an accurate and reliable indication is obtained of the amount of carbon **13** in the enriched material which has entered the sample. This of course is not possible at mass positions 12 and 13 because the intensity of carbon **13** is partially masked by the ion consisting of carbon **12** in combination with hydrogen. In like manner an accurate measure of the enrichment of nitrogen is obtained by comparing the intensity of mass position 7 with 7½, and the measure of the oxygen enrichment is obtained by comparing mass positions 8 and 9. These comparisons are aided by use of the rapid sweep permitted by circuits of the general kind illustrated in Figure 13 which involve rapid scanning.

I claim to have invented:

1. The method of determining the relative abundance of the isotopes of an element in a sample which includes doubly ionizing the atomic material from the sample and measuring the relative intensities of the mass lines at the mass positions corresponding to the doubly charged atomic ions of the isotopes of the element.

2. The method of mass spectrometry which comprises applying sufficient ionizing potential to the ionizing electrons to produce doubly charged ions, effecting separation of the resulting doubly charged ions of different masses from each other and from singly charged ions, and measuring the relative abundance of the different doubly charged ions.

3. A mass spectrometer comprising an ion source including means for increasing the energy of the ionizing electrons to produce doubly charged ions in gases fed thereto, means for separating doubly charged ions of different masses from each other and from singly charged ions, and indicating means for indicating the relative abundance of different doubly charged ions.

4. In combination, evacuated means and a system for feeding gases to said evacuated means comprising conduit means having first and second restrictions for leading the gases to said evacuated means both of which restrictions are much longer than their transverse dimensions and both of which have such large cross section that the flow therethrough is primarily viscous, means for exhausting the conduit means between the two restrictions to a degree of evacuation less than that of the evacuated means, and a fine wire passing through the first restriction.

5. In combination, evacuated means and a system for feeding gases to said evacuated means comprising conduit means having first and second restrictions for leading the gases to said evacuated means both of which restrictions are much longer than their transverse dimensions and both of which have such large cross section that the flow therethrough is primarily viscous, means for exhausting the conduit means between the two restrictions to a degree of evacuation less than that of the evacuated means, and a fine wire passing through the second restriction.

6. In combination, evacuated means and a system for feeding gases to said evacuated means comprising conduit means having first and second restrictions for leading the

gases to said evacuated means both of which restrictions are much longer than their transverse dimensions and both of which have such large cross section that the flow therethrough is primarily viscous, means for exhausting the conduit means between the two restrictions to a degree of evacuation less than that of the evacuated means, and a fine wire passing through both restrictions.

7. The combination of claim 4 having in addition means operable to move one end of the fine wire.

8. The combination of claim 5 having in addition means operable to move one end of the fine wire.

9. The combination of claim 6 having in addition means operable to move the fine wire.

10. In combination, evacuated means and a system for feeding gases to said evacuated means comprising conduit means for effecting viscous flow therethrough and having first and second restrictions for leading the gases to said evacuated means both of which restrictions are much longer than their transverse dimensions and both of which have such large cross section that the flow therethrough is primarily viscous, and means for exhausting the conduit means between the two restrictions to a degree of evacuation less than that of the evacuated means including a pipe having a needle valve therein.

11. The combination of claim 4 having a second fine wire passing through the second restriction.

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