Jan. 9, 1951 C. E. BERRY 2,537,025

MASS SPECTROMETER

Filed April 15, 1946 2 Sheets-Sheet 1

FIG. 1.

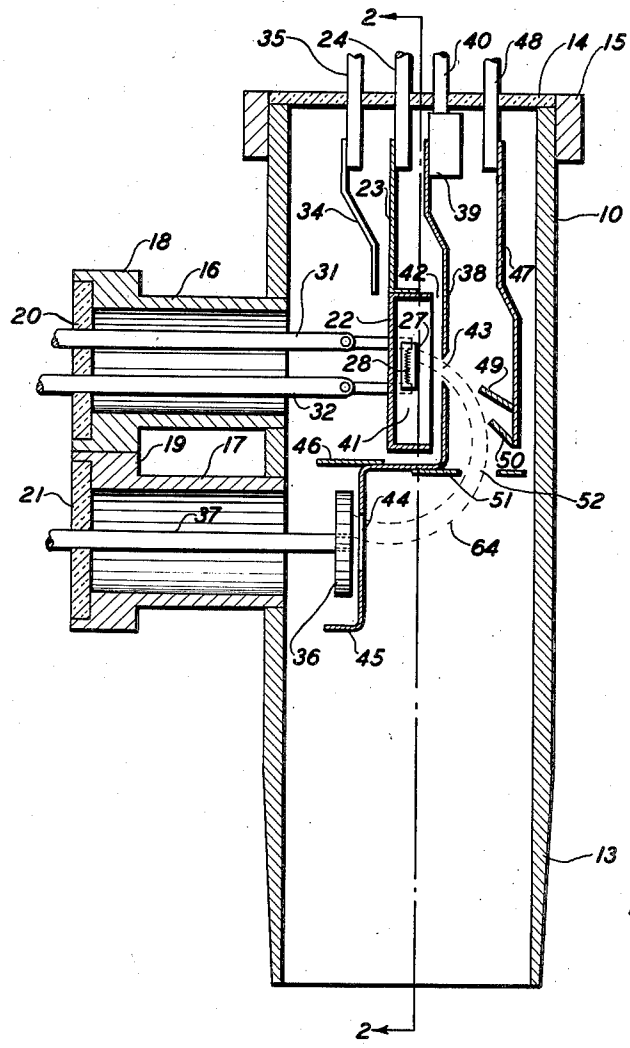

FIG. 2.

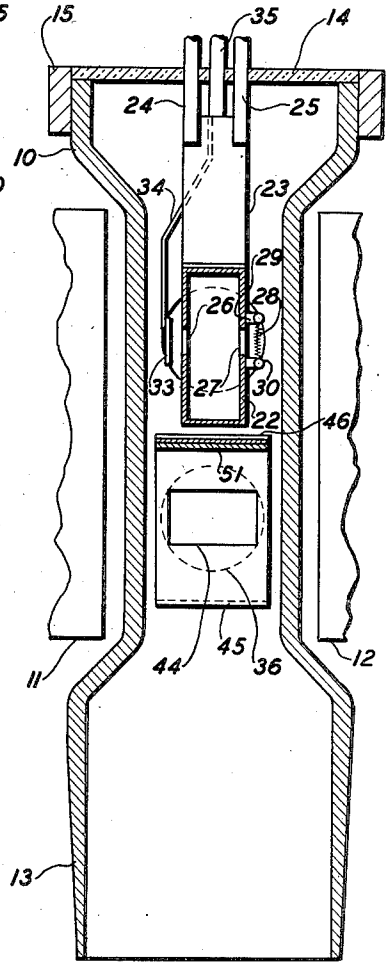

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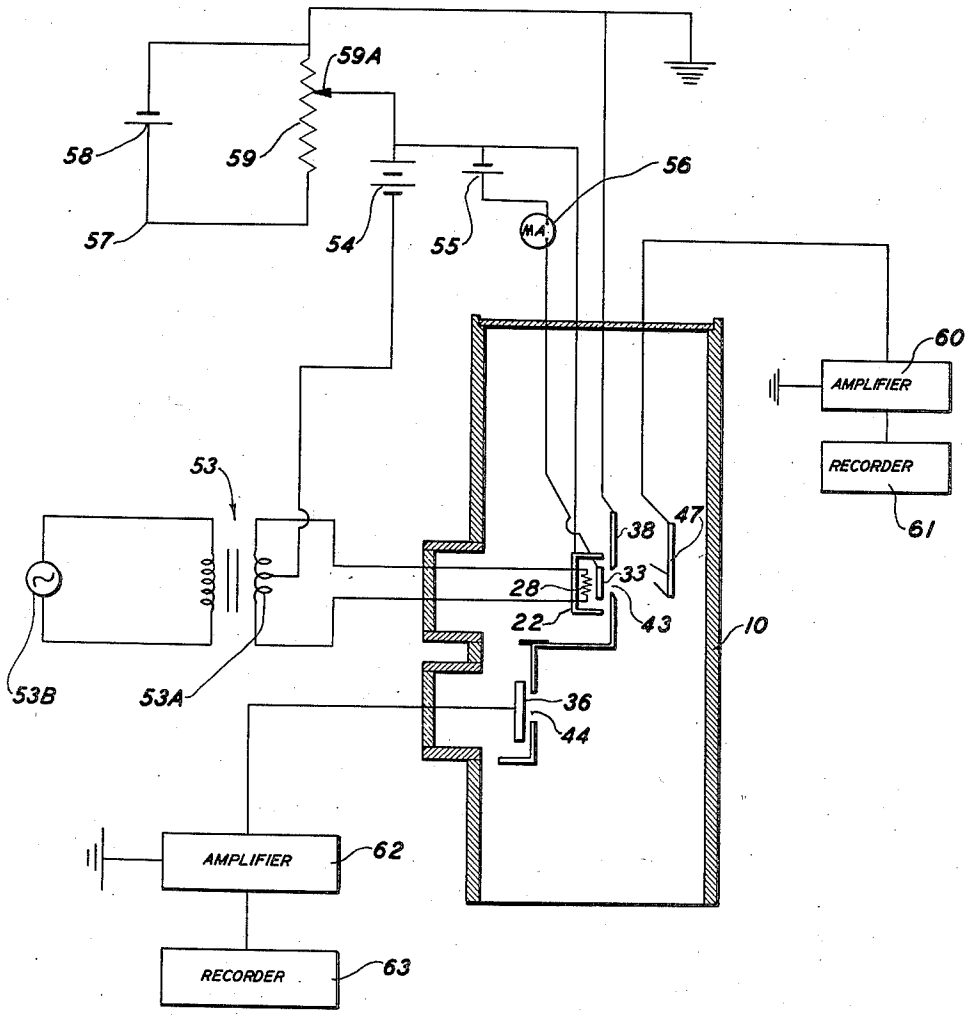
FIG. 3.



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MASS SPECTROMETER

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4 Claims. (Cl. 250—41.9)

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This invention is concerned with mass spectrometry and provides improvements in this field, particularly adapted to the detection of microscopic leaks in high pressure vessels and vacuum systems. In its preferred aspect, the invention contemplates a combined mass spectrometer and ionization gauge.

A mass spectrometer is a vacuum tube device for sorting ions according to their respective mass-to-charge ratios. Molecules introduced into the device are converted into ions, usually by thermal effect or electron bombardment, and the ions thus formed are propelled (usually as a heterogeneous beam) into a magnetic field wherein the several kinds of ions fan out into a set of diverging curved beams, each composed of ions of equal ratio of mass to charge. If one of these homogeneous beams is permitted to impinge and discharge upon a collector electrode, the current thus produced is an index of the relative abundance of this kind of ion in the mixture being analyzed.

It has been proposed previously to detect leaks in pressure vessels and the like by evacuating them and analyzing the gas pumped out. The precise location of a leak in the shell can be found by playing a stream of helium or other gas over the suspected area. When the stream is adjacent the leak the helium will be sucked into the system and can be detected in the stream being evacuated from the vessel. Helium is especially suitable for this purpose because of the ease with which it penetrates even the smallest crack.

A mass spectrometer is a useful instrument for analyzing the gas stream evacuated from a vessel in the foregoing method of leak detection, but those available heretofore have been expensive, complicated and in general too large and insufficiently rugged to meet the requirements of rough field use. There is a distinct need for a small, simple and rugged mass spectrometer for leak detection and similar operations in which it is more important to detect the presence of one or two selected constituents, say helium and nitrogen, than it is to make an accurate quantitative analysis of a mixture having many ingredients, say a gaseous mixture of many hydrocarbons. In other words, a spectrometer to be employed as a leak detector should have the requisites of a portable instrument and at the same time be extremely sensitive to the constituents which indicate the leak. As a result of my investigations, I have developed such a mass spectrometer which, however, may also be adapted to more complicated analyses.

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One of the principal characteristics of the mass spectrometer of the invention is that substantially the entire potential which propels the ions through the magnetic field from ionization chamber to collector electrode is impressed upon the ions in the ionization chamber proper. Thus, in its preferred form, the ionization chamber is formed by a rear vessel or box of conductive material (with its back and preferably its sides as well acting as one propelling electrode) and a front plate, insulated from the rear and provided with a slit through which the ions are propelled with velocity sufficient to reach the collector, by means of a potential impressed between the box and plate portions of the chamber. This structure and method of operation are distinguishable from prior practices in which the ions are accelerated by passing through slits in a plurality of electrodes between which a substantial potential gradient is maintained.

In passing through the slit in the front of the ionization chamber in the preferred spectrometer of my invention, the ion beam fans out under the influence of a magnetic field. Those ions having the proper curvature of path pass through one or more additional slits (in members maintained at substantially the same potential as the front of the ionization chamber and preferably at ground) to a collector electrode, the resulting current being amplified and recorded as a partial spectrum.

If desired, a baffle electrode can be disposed in the analyzer region on the outside of the curved path of the beam of ions (say, helium) passing to the collector. Ions having a greater ratio of mass to charge pursue a straighter path than those passing to the collector and so impinge upon and are discharged at the baffle electrode, the current thus formed being (in many cases) an indication of the relative density of the sample being analyzed. So by connecting an amplifier and a galvanometer to the baffle electrode, the latter serves as an ionization gauge, giving a measure of the partial pressures of all gases present except helium and hydrogen.

The design and principles of operation of the ionization chamber, plus other design features to be discussed hereinafter, permit the construction of a spectrometer of extremely small size as compared to heretofore customary types. Thus the essential parts of the spectrometer proper can all be included within a metallic envelope about 1'' x 1'' x 4'' in overall dimensions. More-

over, these parts are so constructed and arranged that the parts themselves are inexpensive to manufacture and the spectrometer is easily assembled and dis-assembled for cleaning.

By way of example, the ionization chamber enclosure, the electron catcher, the collimator baffles, shields to catch stray ions, the ionization gauge electrode and in fact all of the elements within the spectrometer head may, in accordance with the invention, be mounted on a single detachable member so that all can be withdrawn at once for cleaning or adjustment. I prefer, however, to mount the filament for electron production and the ion collector or target on separate leads projecting through the sides of the envelope.

These and other features of the invention will be understood more thoroughly through reference to the following detailed description, taken in conjunction with the accompanying drawing in which

Fig. 1 is a schematic section through a preferred form of my mass spectrometer taken parallel to the face of the pole pieces of its magnet;

Fig. 2 is a schematic section taken through the spectrometer of Fig. 1 along the line 2—2; and

Fig. 3 is a simplified wiring diagram of the spectrometer of Figs. 1 and 2.

Referring first to Figs. 1 and 2, the spectrometer is enclosed in a metallic envelope 10, preferably one made of oxygen free high conductivity copper. This may be made conveniently from a piece of copper tubing having about 1 inch inside diameter and which is flattened into rectangular cross section between its two circular end portions to accommodate the poles 11, 12 of a permanent magnet of "Alnico" or the like. The poles are placed close to the rectangular portion so that the lines of force run through the entire interior of the rectangular portion of the envelope, i. e. through the regions in which ions are formed and also those in which sorting occurs. The lower portion 13 of the tube is tapered on the outside to fit a standard tapered joint for connection to a vacuum system (not shown). The upper end of the envelope is sealed by a circular glass plate 14 which fits within a copper flange or ring 15 that is welded to the outside of the envelope. Two copper nipples 16, 17 provided with flanges 18, 19 project into the envelope at right angles to its major axis (from the left side as shown in Fig. 1). These likewise are sealed respectively by insulating plates 20, 21.

Within the envelope at approximately the level of the upper nipple is a box 22 which is rectangular in both plan and elevation and stands vertically with its front open (and facing the right in Fig. 1). It is held in place by an angle piece 23 welded to its top and extending upward to one or more terminal posts 24, 25, which project through seals in the glass plate 14 (see Fig. 2). The box, like most of the other metal parts within the envelope, preferably is made of "nichrome" or equivalent. The box has two matching rectangular slits 26, 27 in its sides to provide passage for an electron beam through the box transverse to the direction of ion propulsion.

The electrons are provided by a filament 28 mounted on a pair of L-shaped horizontally placed conductive posts 29, 30 which in turn are mounted respectively on leads 31, 32 which pass out of the envelope through the upper nipple and its sealing plate 20. The filament is mounted outside the box in a vertical position opposite one of the slits or windows. Outside the opposite side of the box an electron catcher 33, say a small gold plate, is mounted and held in position by a lead 34 that passes upward to another terminal post 35 that passes out through a seal in the glass plate 14. As described in detail hereinafter, an electron beam is formed when the filament is heated and potentials are applied to cause the electrons to pass through the box from the filament to the catcher.

A collector electrode 36 is mounted concentrically on the end of a conductive post 37 that passes out through the sealing plate 21 in the lower nipple 17, the post being horizontal and the collector (which is a circular disc) being mounted with its major surfaces vertical.

A combined electrode and shield 38, which performs a multiplicity of functions, is suspended from a mounting block 39 which in turn is fastened to one or more terminal posts 40 that pass up through seals in the glass plate 14 at the top of the envelope. The combined electrode and shield (hereinafter called a "composite electrode") is made of Nichrome sheet and passes in front of and covers but does not seal the box (which forms the rear of an ionization chamber 41), being separated from the front of the box by a small dielectric gap 42. A rectangular ion slit 43 is cut horizontally in the composite electrode about midway of the box, the sides of the slit being parallel and beveled (as shown in Fig. 1) to knife edges at the rear face.

Immediately below the box, which encloses all but the front of the ionization chamber, the composite electrode is bent horizontally to the left and then vertically again to pass in front of the collector electrode. An opening 44, preferably of rectangular shape, is provided in the composite electrode to permit ions to pass to the collector. The opening is smaller than the collector, and the latter is shielded from vagrant ions by means of a lower shield 45 formed as a flange bent horizontally to the left on the bottom of the composite electrode and a matching horizontal upper shield 46 of sheet metal welded to the composite electrode at the bend just above the collector.

An ionization gauge electrode or baffle 47 is suspended from a terminal 48 mounted in a seal in the glass plate 14 at the top of the envelope. This baffle is in plate form and hangs in front of the composite electrode and generally parallel to it. On its lower portion it bears two projecting plates 49, 50 which extend toward the composite electrode obliquely to the major surface of the baffle and roughly tangential to the paths of ions of high mass-to-charge ratio issuing through the slit. It collects and discharges such ions.

If desired, a collimator 51 may be provided in the ion path from ionization chamber to collector. Conveniently this is placed horizontally and welded to the composite electrode, extending from it toward the right with a slit 52 for the passage of ions to the collector.

It will be observed that the slit in the collimator is wider than that in the front plate of the ionization chamber, and that the opening in front of the collector or target is still wider. This is desirable in that it facilitates collection of all helium or other "detector" ions and thus increases sensitivity.

In considering the energization of the spectrometer, reference should be made to Fig. 3, which shows the device of Figs. 1 and 2 schematically, like parts being indicated with reference numbers of Figs. 1 and 2.

The filament is energized with alternating current supplied from a conventional A. C. source 53B through a filament transformer 53. The center of the output coil 53A of the transformer is connected to the box 22 which forms the back of the ionization chamber through a direct current source 54. The latter supplies the accelerating voltage which causes the electrons to pass as a beam into the box. A second direct current source 55 is connected between the first source 54 and the electron catcher 33 through a milliammeter 56. The second source maintains a proper potential gradient from box to catcher to cause the beam to pass out of the box to the catcher. In other words, the battery 55 supplies potential to the catcher, making it positive with respect to the box and still more positive with respect to the filament.

The sensitivity of the spectrometer is proportional to the current passing through the milliammeter, which is used for adjustment purposes.

The box is also connected in another circuit with a potential supply network 57 comprising a direct current source 58 in series with the ends of a potentiometer 59. The slider 59A of the potentiometer is connected to the box, the end of the potentiometer being connected through the network to ground and to the composite electrode.

The ionization gauge electrode is connected to ground through a vacuum tube amplifier 60 which is in turn connected to a conventional galvanometer 61, the combination of baffle, amplifier and galvanometer serving as an ionization gauge.

The collector electrode is likewise connected to ground through a vacuum tube amplifier 62 which has an associated galvanometer 63 that serves to indicate the mass spectrum.

The network 57 supplies ion accelerating voltage between box and composite electrode, these two serving as the sole ion accelerating electrodes of the spectrometer. The box, it will be observed, also serves as an electrode in the electron acclerating circuit.

To consider the operation of the device, let it be assumed that a pressure vessel is being tested for leakage by placing it under a high vacuum and playing a stream of helium progressively over various parts of the outside surface of the vessel, so that if there is a leak (even one of very small dimension) the helium will pass through and almost instantly diffuse through the pumping system used to evacuate the vessel.

The mass spectrometer has been connected to the pumping system at the start of the operation and its interior is, in effect, a part of the system. So the helium molecules diffuse into the ionization chamber of the spectrometer together with molecules of other gases present, usually those which art the major constituents of air, i. e. nitrogen and oxygen.

The action of the spectrometer is as follows:

Gas molecules in the ionization chamber are bombarded by electrons passing through it from the window 27 to the window 26 and are thus ionized. The resulting ions are propelled out of the ionization chamber through the slit 43 by a potential impressed by the network 57 between the box 22 or rear ion accelerating electrode and the composite electrode 38, which acts as the other ion accelerating electrode. Thus a heterogeneous beam of ions having various mass-to-charge ratios is formed, but immediately begins to separate, even in the ionization chamber, which lies within the field of the magnet. Ions of nitrogen and oxygen have a much greater mass than those of helium and hence pursue paths having a much larger radius than helium ions.

The geometry of the device and the potentials applied are such that the helium atoms pursue approximately the circular path 64 indicated on Fig. 1, and passing through the slot 52 in the collimator 51 and the hole 44 in front of the collector, impinge upon the latter and are discharged to produce a current that is indicative of the amount of helium present.

Simultaneously the ions of greater mass, and hence of greater mass-to-charge ratio, for example ions of oxygen and nitrogen, are propelled along paths of greater radii and are trapped when they strike the ionization gauge electrode 47 with its projections 49, 50. These ions likewise produce a current, which is roughly indicative of the total amount of gas present.

For leak detection, employing helium, the spectrometer illustrated may be so proportioned that an ion accelerating potential of only about 25 volts (impressed across the ionization chamber) will cause the helium ions to pursue a curved path with a radius of about 0.4 inch, to lodge at the collector. Since the radii of the paths of the major gaseous constituents of air are much greater, it is not difficult to adjust the position of the ionization gauge electrode to bring about a clean separation. Since ordinarily the ions of nitrogen, oxygen, et cetera caught on the baffle electrode will be many times more numerous than those of helium, say in a ratio of 50,000 to 1, the current collected at the ionization gauge electrode will be closely proportional to the total ions, hence its usefulness as an ionization gauge.

By adjusting the slider of the potentiometer, the position of the entire fan of ion beams issuing from the ionization chamber may be adjusted, and in this way the helium beam 64 can be focussed on the collector electrode.

It should be noted that all three apertures 43, 52, 44 through which the ion beam passes are at the same potential, in this case at that of ground, so that vagrant ions are collected on the plates that define these apertures and do not cause interference with the helium spectrum.

As I have already indicated, the particular example of the spectrometer illustrated in the drawings is designed for leak detection employing helium in air. Consequently, it is not necessary to employ a beam sweeper circuit (such for example as that illustrated in application Serial No. 499,055, filed August 18, 1943, now Patent No. 2,397,560). However, spectrometers of the type of the invention may also be employed for quantitative and qualitative analysis of gaseous mixtures containing many components, in which case a beam sweeper circuit might well be employed, although the same result could be obtained by manually regulating the ion accelerating voltage by varying the position of the slides of the potentiometer or by varying the current input to an electromagnet substituted for the permanent magnet shown.

It is not essential to employ a metallic envelope, but it is highly desirable since it adds greatly to the ruggedness of the apparatus and also provides additional surface for discharging vagrant ions.

The conditions of service of a mass spectrometer employed for leak detection are much more severe than in the case of the ordinary laboratory apparatus, not only from the standpoint of

physical shock, but also from the standpoint of the amount of matter accumulated on the inner surfaces of the apparatus as occluded films, et cetera.

The spectrometer illustrated is excellently adapted to this service, because it is easy to disassemble and clean. All of the working parts except the filament and the collector are attached to the glass insulator plate 14 on the top of the envelope. It is merely necessary to unseal this plate (which conveniently is held with wax or the like) in order to withdraw the bulk of the working parts together for cleaning. The collector electrode and the filament can also be withdrawn through their respective nipples and it is the work of but a few minutes to completely disassemble, clean, and reassemble the entire spectrometer.

The parts of the spectrometer are simple to fabricate, and the composite electrode with its attachments, such as the shields and the collimator serves a number of functions, being at once one electrode in the ion accelerating system of the ionization chamber, a collimator for the beam passing to the collector, and a shield for grounding vagrant ions.

Because of the construction and arrangement of its elements, the spectrometer of the invention can be very small, with the result that the ion accelerating voltage is of the order of 25 volts, instead of the 4000 volts heretofore employed. This is an important safety feature, especially in a portable instrument for use in the field.

The spectrometer of the invention, even taking into account the associated amplifiers, meters, et cetera, can be built for but a small fraction of the cost of heretofore customary mass spectrometers, including prior simplified versions designed for leak detection. This, combined with inherent safety of operation, rugged design, easy accessibility for cleaning, high sensitivity and small size and weight make it an outstanding instrument for both field and laboratory in cases where a high degree of resolution is not required.

I claim:

1. In a mass spectrometer the combination which comprises a tubular envelope, sealed at one end with an insulating plate and open at the other end for connection to an evacuating system, an ionization chamber having a rear wall of conductive material, a front conductive wall in the chamber for an ion slit and insulated from the other walls of the chamber, the ionization chamber receiving molecules of gas to be ionized by diffusion from the open end of the envelope, a magnet between the poles of which the ionization chamber is disposed, means for producing a potential between the front and rear walls of the ionization chamber to cause ions formed in the ionization chamber to be expelled through the ion slit into the space between the two poles of the magnet, whereby they pursue a curved path between the poles, a collector electrode disposed in the path of the ions between the two poles, a shield disposed in the path of the ions in front of the collector electrode and provided with an opening for ions which is wider than the slit in the front wall of the ionization chamber, and a collimating baffle disposed between the shield and the front wall and provided with an orifice for the passage of ions which is intermediate in width between the opening and the shield and the slit in the front wall of the ionization chamber.

2. In a mass spectrometer the combination which comprises a tubular envelope, sealed at one end with an insulating plate and open at the other end for connection to an evacuating system, an ionization chamber having a rear wall of conductive material, a front conductive wall on the chamber for an ion slit and insulated from the other walls of the chamber, the ionization chamber receiving molecules of gas to be ionized and analyzed by diffusion from the open end of the envelope, a magnet between the poles of which the ionization chamber is disposed, means for producing a potential between the front and rear walls of the ionization chamber to cause ions formed in the ionization chamber to be expelled through the ion slit in the space between the two poles of the magnet whereby they pursue a curved path between the poles, a collector electrode disposed in the path of the ions between the two poles, a baffle having a slit disposed in the path of the ions to the collector electrode, and means for maintaining both baffle and front wall at substantially ground potential.

3. A mass spectrometer according to claim 2 in which the tubular envelope is of oxygen free copper.

4. In a mass spectrometer the combination which comprises a tubular envelope sealed at one end with an insulating plate and open at the other end for connection to an evacuating system, an ionization chamber having a rear wall of conductive material, a front conductive wall in the chamber having an ion slit and insulated from the other walls of the chamber, the ionization chamber receiving molecules of gas to be ionized and analyzed by diffusion from the open end of the envelope, a magnet disposed on the outside of the envelope between the poles on which the ionization chamber is disposed, means for producing a potential between the front and rear walls of the ionization chamber to cause ions formed in the ionization chamber to be expelled through the ion slit into the space between the two poles of the magnet whereby they pursue a curved path between the poles, a collector electrode disposed in the path of the ions between the two poles, a baffle having a slit disposed in the path of the ions to the collector electrode, means for maintaining the baffle and the front wall at substantially the same potential, a baffle electrode disposed between the baffle and the front wall on the outside but adjacent to the curved path of the ions, and current indicating means connected to the baffle electrode.

CLIFFORD E. BERRY.

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