

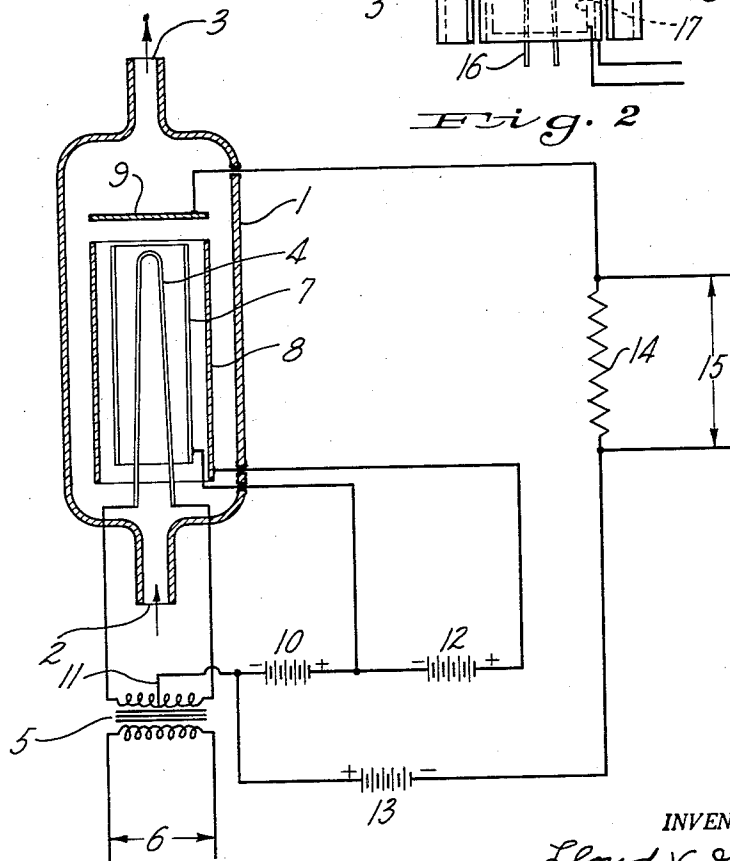
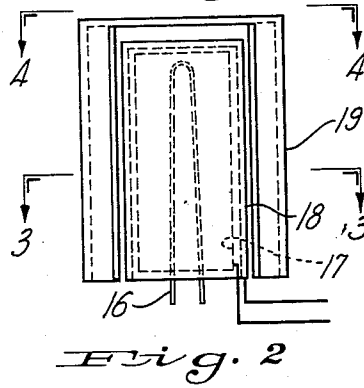
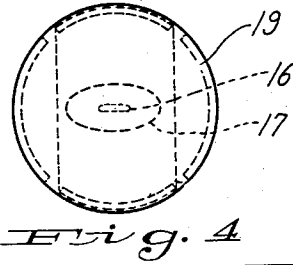
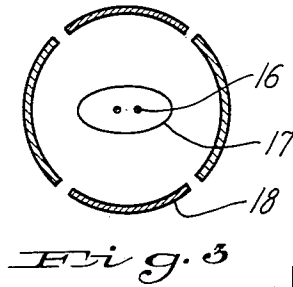
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L. V. GUILD

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GAS PHASE DETECTION BY ELECTRON EMISSION AND IONIZATION

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INVENTOR.

Lloyd V. Guild
BY William B. Jaspart
Attorney.

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3,149,279 GAS PHASE DETECTION BY ELECTRON EMISSION AND IONIZATION

Lloyd V. Guild, Pittsburgh, Pa., assignor to Burrell Corporation, Pittsburgh, Pa., a corporation of Pennsylvania
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This invention relates to new and useful improvements in gas chromatography, more particularly to means for detecting small quantities of gas phase material in the order of their occurrence in an elongated column packed with either a solid adsorbent or an inert material coated with a high boiling liquid and continuously flushed with an inert gas such as helium or nitrogen.

The sample to be analyzed, either liquid or gas, is injected into the inert gas stream and washed through the column in the gas phase, each component of the mixture passing through the column in a time dependent upon the molecular affinity of the component for the packing. The detector must be able to pick out the components from the carrier gas stream as it emerges from the end of the column mixed with the carrier gas.

Ionization has been shown to be a possible means for accomplishing this detection. Thus, when a gas atom looses an electron, a positively charged ion is formed and this positively charged ion is capable of conducting electrical current and advantage can be taken of this to make a gas detector. Bombardment of the gas molecules by radioactive means has been used with strontium 90 or radium to effect the ionization. Radio frequency energy has been employed to achieve ionization and the use of electron emission has also been suggested as a means of achieving ionization.

In accordance with the present invention, I employ an electron tube generator which is capable of providing high electron density from which a high percentage of ionization occurs.

It is among the objects of this invention to provide an emission type ionization detector employing a grid, a filament and plate in which the plate is placed at right angles to the path of electrons emitted by the filament and passed through the grid.

It is a further object of this invention to provide an emission type ionization detector comprising a grid, filament and a concentric plate having a positive potential to generate high electron density and a collector plate at right angles.

It is yet another object of this invention to provide such electron tube generator with thoriated iridium filaments and with rhenium filaments. These and other objects of the invention will become more apparent from a consideration of the accompanying drawing constituting a part hereof in which like reference characters designate like parts and in which:

FIGURE 1 is a diagrammatic illustration of an electron tube generator with a power and measuring circuit embodying the principles of this invention;

FIGURE 2 is a modified form of the tube of FIGURE 1;

FIGURE 3, a plan view of a grid filament and plate taken along the line 3—3, FIGURE 2; and,

FIGURE 4, a section taken along the line 4—4, FIGURE 2.

With reference to FIGURE 1 of the drawing, the detector is housed in a container of glass or metal, as shown at 1, with a gas inlet 2 and an outlet 3. A filament 4, preferably of thoriated iridium or rhenium, is heated by a step-down transformer 5 from a potential source applied at 6. The filament 4 is surrounded by a cylindrical or elliptical shaped grid 7 and a cylindrical or elliptical plate 8 and a disc-shaped plate 9, which latter

is at right angles, as shown. The filament 4 and grid 7 and the plates 8 and 9 are provided with appropriate leads or electrical conductors, as shown. A potential is applied from a source 10 between the center tap 11 of the transformer 5 and the grid 7 so that the filament 4 is negative with respect to the grid 7 and a potential is applied from a source 12 between the grid 7 and plate 8 such that plate 8 is positive with respect to the grid 7 and more positive than the grid 7 with respect to the filament 4. A potential is applied between the filament 4 and plate 9 and the pressure reduced. If the filament 4 is heated hot, electron emission occurs. The electrons are drawn toward the grid 7 which serves as an accelerator and most of them pass through the grid and are attracted to the more positive plate 8 so that a high electron density exists between the filament 4, grid 7 and the plate 8 and a high percentage of ionization occurs. By applying a potential from a source 13 between the filament 4 and plate 9 with resistor 14 in series with the potential source 13 and the plate 9 is negative with respect to filament 4, ions that are formed will be swept by the gas stream and attracted by the highly negative charged plate 9. Because plate 9 is at right angles to the filament 4 and the grid 7, no electron repression occurs and a very high potential can be applied of the order of several hundred volts. This varies somewhat with pressure, but with ordinary pressures, voltages of the order of 300 to 500 volts can ordinarily be applied. When ions are formed, current flows between the filament 4 and plate 9 giving a potential drop across resistor 14 which can be measured by any suitable potential measuring device at 15. Also, battery 12 may be omitted and plate 8 connected to grid 7.

By using the above-described device, the difficulty of low electron density is overcome by utilizing the additional plate 9 at right angles to yield high electron density. The difficulty of electron repression is also overcome by placing the additional plate 9 at right angles to the system. The probability of ion formation is a function of the concentration of the ionizable material.

In the modified tube shown in FIGURES 2, 3 and 4 of the drawing, increased sensitivity is obtained by plate structure which will give a higher plate collecting surface. The numeral 16 designates a filament and 17 an electrical grid. The numeral 18 designates a split plate which is placed outside the grid along the axis. This is more clear from FIGURE 3 where only the grid 17, filament 16 and plate 18 are shown. In FIGURE 4, 19 designates a collector plate. Plate 18 is not shown, but filament 16, grid 17 and plate 19 are shown. The collector plate 19 is of cylindrical shape with two segments of the circle extending downward parallel to the grid and opposite the small axis of the elliptical grid 17. The plate 18 is not absolutely necessary and may be eliminated with some reduced performance in any of the modifications described. Also, it is possible to eliminate the grid 17 and place a positive potential directly on the plate 18 with some reduction in performance.

From the foregoing, it is evident that it is possible to use very high detector plate voltages and still operate at any desired ionization potential. The ionizing ability of the tube is a function of the energy of the electrons themselves, the energy being controlled by the potential difference between the filament 16 and the grid 17 and plate 18. When either the grid 17 or plate 18 are eliminated, it becomes a function of the potential difference between the filament 16 and the other element. The potential on the plate 19 controls the attraction of the plate for the ions and by locating at right angles, or as shown in FIGURE 2, it does not in any way affect the energy of the electron. Thus it is possible to operate below the ionization potential of the carrier gas and yet

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achieve a high collection efficiency for the materials in the carrier having a lower ionization potential and give a high signal. By isolating the collector plate as described, the device is very little affected by electrical noise from the filament.

Although one embodiment of the invention has been herein illustrated and described, it will be evident to those skilled in the art that various modifications may be made in the details of construction without departing from the principles herein set forth.

I claim:

1. Apparatus for detecting small quantities of gas phase material comprising an electron tube generator in the shape of a housing having an inlet and outlet for passing gases therethrough, a filament disposed in the path of the gases flowing through said housing, said filament being connected to a source of electrical power, a grid disposed around said filament, a plate surrounding said grid, and a second plate disposed transversely of and in the path of the gas flow, said grid and plates being connected in an electrical circuit having means for measuring the relative current flow for the different gases passing through said generator.

2. Apparatus for detecting small quantities of gas phase material comprising an electron tube generator in the shape of a housing having an inlet and outlet for passing gases therethrough, a filament disposed in the path of the gases flowing through said housing, said filament being connected to a source of electrical power, a grid disposed around said filament to accelerate the electrons given off by said filament, a plate surrounding said grid

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for collecting said electrons, and a second plate disposed transversely of and in the path of the gas flow, said grid and plates being connected in an electrical circuit having means for measuring the relative potential drop for the different gases passing through said generators.

3. Apparatus as set forth in claim 1 in which the first plate is concentric with the grid and has a positive potential and the second-named plate is disposed at right angles to said first-named plate and has a negative potential.

4. Apparatus as set forth in claim 1 in which the filament is thorium coated iridium.

5. Apparatus as set forth in claim 1 which employs a rhenium filament.

6. Apparatus as set forth in claim 1 in which the first-named plate is a split member and the collector plate is of cylindrical shape.

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