United States Patent

[72] Inventors Ralph Elliott Mayo Fairfield; Edward B. Delany, Ridgefield, Conn. Appl. No. [21] 692,884 [22] Filed Dec. 22, 1967 [45] Jan. 19, 1971 Patented [73] Assignee **The Perkin-Elmer Corporation** Norwalk, Conn. a corporation of New York [54] ION SOURCE FOR A MASS SPECTROMETER 10 Claims, 10 Drawing Figs. [52] U.S. Cl. 250/41.9 [51] Int. Cl...... H01j 37/08 Field of Search...... 250/41.9ISB, [50] 49.53, 41.9ISE; 313/83

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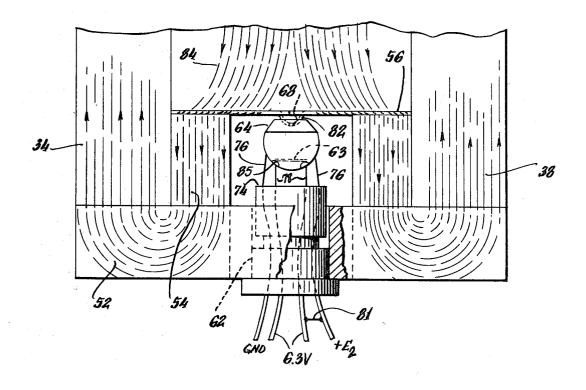
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Primary Examiner-James W. Lawrence			

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Assistant Examiner—C. E. Church

Attorney-Edward R. Hyde, Jr.

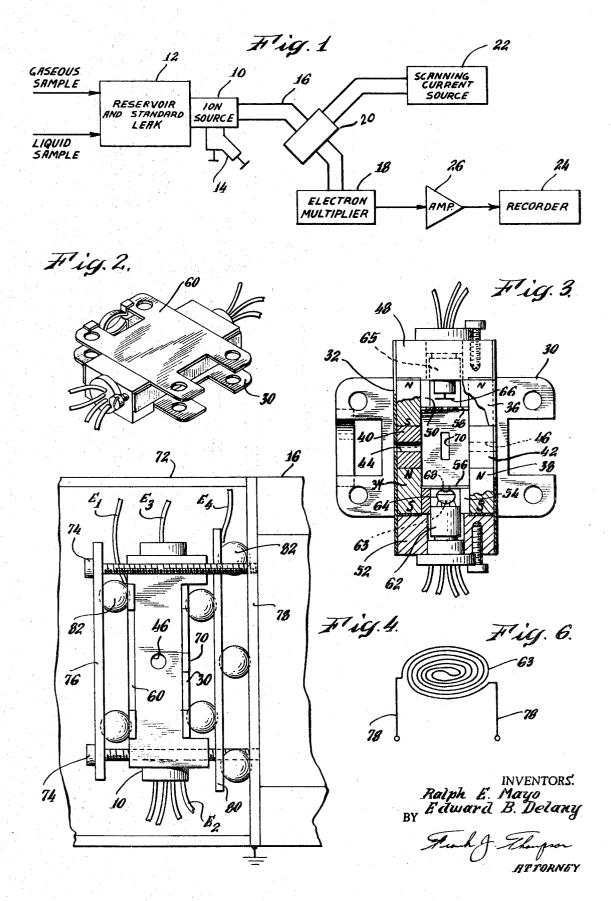
ABSTRACT: An ion source includes a directly heated cathode formed of an elongated wire arranged in a configuration for providing a relatively large electron emission surface. The cathode is surrounded by a grid electrode having a hollow semispherically shaped segment of radius R_1 and a frustum conically shaped segment. A first apertured anode electrode is positioned with respect to the grid electrode and includes a spherical segment extending toward the grid electrode and shaped as a hollow apertured spherical body of radius R_2 , where $R_1 > R_2$. With this arrangement a relatively high emission current density is provided while electrons are focused through the first anode aperture with a relatively noncomplex structure.



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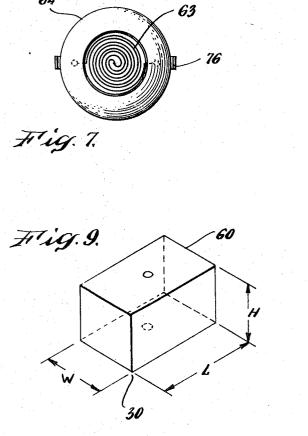




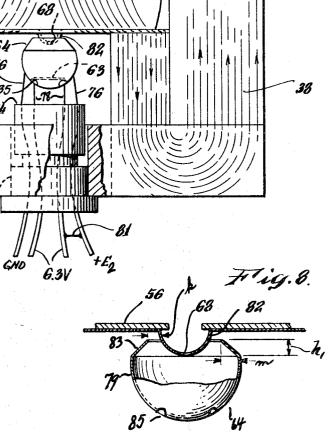
Fig.5.

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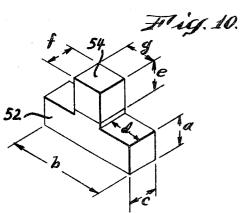
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INVENTORS, Ralph E. Mayo BY Edward B. Delany

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ION SOURCE FOR A MASS SPECTROMETER

This invention relates to electron-optic arrangements. The invention relates more particularly to an electron-optic arrangement for an ion source of a mass spectrometer.

An ion source for a mass spectrometer is adapted for creating a plurality of ion constituents from a sample by electron bombardment of vaporized sample molecules. The resultant ions are accelerated toward an ion target electrode in an analyzer section of the spectrometer and travel through a 10 time-varying magnetic field. This field is adapted for focusing ions of differing m/e ratios at an aperture of the target electrode. Ions passing through the ion target aperture excite an electron multiplier and an output signal thereof is coupled to a recording means for recording the mass spectrum of the sam- 15 ple.

In addition to a cathode source of electrons and an electron target electrode, an ion source includes electron-optic means. A pair of spaced-apart apertured plates are disposed in the electron flow path and define wall sections of an ionization 20chamber. One of these plates functions as a first anode and, in cooperation with a grid electrode maintained at cathode potential, operates to focus the electrons at an aperture in its cross section. Magnetic field generating means are also provided for establishing an axial electron beam collimating field. This arrangement provides a relatively narrow electron beam for impacting sample molecules in the ionization chamber.

The sensitivity of a mass spectrometer is dependent largely upon the efficiency with which the ion source creates ions from the sample molecules. At relatively low sample leak rates and in those instances when the sample quantity is limited, the ion source efficiency can be determinative of a meaningful analysis.

Ions are created by the ion source when the vaporized sample molecules drift into the path of the electron beam in the ionization chamber. The resulting particle collisions, as well as secondary collisions between electrons, ions, and sample molecules produce ions. Electron beam intensity is an important factor in providing efficient operation of the ion source. 40The probability of electron-molecule collision and the ion source efficiency generally increases with electron beam intensity. It is thus advantageous to provide an ion source which generates a relatively high beam current.

The cathode electron source generally comprises a directly 45 heated cathode having a hairpin shape and is surrounded by a grid electrode of cylindrical shape. Although from an electron-optical viewpoint, the hairpin arrangement approximates a point source and facilitates electron focusing, the electron emission capacity of such a hairpin cathode is limited to a 50 value below electron current densities desirable for providing increased ion source efficiency. Cathode emission capacity can generally be increased by operating the source at a relatively higher temperature. However, an undesirable shortened cathode life accompanies the higher temperature operation.

In an alternative electron source for providing increased electron emission, a relatively larger emission surface is provided. Because of the relatively small dimensions of an ion source, electron focusing with a cathode emission surface of increased physical size is rendered difficult. In one arrange-60 ment for focusing electrons from a relatively large cathode surface, a hollow hemispherical grid electrode, i.e., a grid electrode shaped as one-half of a hollow sphere, is positioned about the cathode near a dimpled aperture segment of the first anode. The aperture is relatively small, and it is found that 65 although this arrangement accommodates a relatively larger electron current, the current is limited by virtue of the aperture size to current densities less than desired. As the aperture is enlarged to accommodate larger current densities, the focusing field is seriously distorted.

A modification to this electrode arrangement adapted for a larger aperture size and corresponding current densities includes auxiliary field shaping electrodes spaced between the hemispherical electrode and dimpled first anode. However, such a modification results in a relatively complex electrode 75

structure and introduces structural and fabrication problems with respect to mounting, spacing, and insulation of the electrodes.

Accordingly, it is an object of this invention to provide an improved ion source for a mass spectrometer.

Another object of the invention is to provide an ion source of relatively high current density.

Another object of the invention is to provide an improved electron-optical arrangement with relatively large cathode electrode.

The magnetic circuit of prior ion source arrangements functioned with a hairpin cathode to additionally and beneficially enhance the electron focusing characteristics at the first anode electrode. In this regard, the axial lines of flux extend to the vicinity of the cathode electrode and exert a collimating force on the electrons outside of the ionization chamber. In an ion source having a relatively large cathode, substantial collimating outside the ionization chamber is disadvantageous since it hinders the desired electron focusing.

A further object of the invention is to provide in an ion source an improved magnetic circuit for use with a cathode having a relatively large emission surface area.

In accordance with the present invention, an ion source includes a directly heated cathode formed of an elongated wire 25 arranged in a configuration for providing a relatively large electron emission surface. The cathode is surrounded by a grid electrode having a hollow hemispherically shaped segment of radius R1 and a hollow frustum conically shaped segment. A first apertured anode electrode is positioned with respect to 30 the grid electrode and includes a segment extending toward the grid electrode and shaped as a hollow apertured spherical segment of radius R_2 , where $R_1 > R_2$. With this arrangement a relatively high emission current density is provided while electrons are focused through the first anode aperture with a rela-35 tively noncomplex structure.

In accordance with another feature of the invention, a magnetic circuit for the ion source includes the first anode electrode formed of a ferromagnetic material. The magnetic flux is thereby substantially diverted by the first anode about the grid electrode and the cathode electron source to thereby substantially avoid interference with the electrostatic focusing.

These and other objects and features of the invention will become apparent with reference to the following specifications and drawings wherein:

FIG. 1 is a diagram illustrating the general arrangement of a mass spectrometer;

FIG. 2 is a perspective view illustrating the general arrangement of an ion source constructed in accordance with the present invention:

FIG. 3 is a view, partly cut away and partly in section, of the ion source of FIG. 2;

FIG. 4 is a side view of the ion source of FIG. 2 assembled with ion accelerating electrodes of the mass spectrometer;

FIG. 5 is an enlarged view of the electron source of FIG. 3;

FIG. 6 is a perspective view of the cathode of FIG. 5;

FIG. 7 is a plan view of the grid electrode and cathode of FIG. 5:

FIG. 8 is a more detailed view of the first anode and grid electrodes of FIG. 5; and

FIGS. 9 and 10 are views simulating ion source members and presented for indicating dimensional characteristics of a particular embodiment of the invention.

In FIG. 1, the general arrangement of a mass spectrometer incorporating features of the present invention is shown. The ion source represented generally as 10 receives a gaseous sample or a vaporized liquid sample from a reservoir and standard leak 12. Alternatively a solid sample is vaporized and introduced to the ion source by a solid sample injection probe

14. An electron beam of the ion source creates positive ions by collision with sample molecules and these ions are accelerated over a confined path defined by a conduit 16 toward an ion target electrode and electron multiplier 18. These ions traverse a time-varying magnetic field which is established by

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an electromagnet 20 and a source of scanning current 22. An output signal from the electron multiplier 18, having an amplitude which is representative of the abundance of ions of a particular m/e number, is applied to a recorder 24 by an amplifier 26.

The ion source 10 is illustrated in greater detail in FIGS. 2 and 3. A first stainless steel support plate 30 has mounted thereon a plurality of permanent magnets 32, 34, 36, and 38. The magnets 32 and 34 are separated by a pole piece 40 and the magnets 36 and 38 are separated by a pole piece 42. The pole pieces 40 and 42 include channels 44 and 46 respectively extending therethrough for introduction of a vaporized sample from the standard leak 12 at channel 44 or, the introduction of a solid sample from injection probe 14 at channel 46. A magnetic circuit is formed by the magnets and pole pieces along with the pole pieces 48 and 50, positioned at one end of the ion source, and pole pieces 52 and 54 and a first anode 56, positioned at an opposite end of the ion source. The elements 48-56 are formed of a ferromagnetic material such as cold rolled steel. An ionization chamber is defined by the assembly of the magnets 32, 34, 36, and 38, the pole pieces 44 and 46, the first anode 56, an apertured plate 58 formed of ferromagnetic material, the support plate 30, and a second stainless steel support plate 60 (FIG. 2). The pole pieces 48, 50, 52, 25 and 54 include a cylindrical bore extending therethrough. A ceramic header 62 supports a directly heated rhenium cathode electron source 63 and a stainless steel grid electrode 64 within the bore formed by pole pieces 52 and 54. Similarly, a ceramic header 65 supports a stainless steel or gold plated 30 electron target 66 in the bore formed by pole pieces 48 and 50. Electrons emitted by the cathode 63 are focused through aperture 68 of the first anode 56; are collimated by a magnetic field established by the referred-to permanent magnets into a relatively narrow beam; and are accelerated toward the elec- 35 tron target electrode 66. Electrons colliding with molecules in the ionization chamber create positive ions which are accelerated into the analyzer section of the spectrometer through an aperture 70 in the lower support plate 30.

FIG. 4 illustrates an assembly of the ion source and ion ac- 40celerating electrodes. The ion source 10 is mounted in an evacuated chamber 72 by screws 74 extending through holes in a metal mounting plate 76 and mating with threaded holes in a metal analyzer section entrance plate 78. These screws secure together the ion source 10, the plates 76 and 78, a 45 metal ion accelerating plate 80, and a plurality of ceramic insulating spheres 82 which extend into indexing apertures located in these plates. In one arrangement the plates 76 and 78 are maintained at ground potential while a relatively positive potential E_1 is applied to the ion source assembly 10 via plate 60. The ion source assembly is thereby maintained at the potential E_1 . A positive potential E_2 is applied to the cathode and grid electrodes which are insulated from the assembly by the ceramic header 62 and a positive potential E_3 is applied to 55 the electron target electrode which is similarly insulated from the ion source assembly by ceramic header 65. A positive ion accelerating potential E4 is applied to the accelerating electrode 80. Positive ions exit from the ion source at aperture 70, pass through a slot in the accelerating electrode 80 and enter $_{60}$ the analyzer through an aperture in the entrance plate 78.

FIGS. 4, 5, 6, 7, and 8 illustrate in greater detail the electron source and an electron-optic arrangement constructed in accordance with features of the present invention. The header 62 supports grid electrode 64 which is spaced from a surface 65 effective electron emission surface of approximately 7.5×10^5 74 of the header by rigid platinum support leads 76. The grid electrode includes a hollow semispherically shaped segment 79 (FIG. 8) of radius R₁, and a frustroconically shaped segment 83 of length h₁. These characteristics are illustrated in detail in FIG. 8. The directly heated cathode electrode 63 is 70 supported on the header and within a contained volume of the grid electrode 64 by leads 78. These cathode leads extend through apertures 85 formed in a lower surface of the semispherically shaped segment 79. The cathode electrode configuration is shown in more detail in the perspective view of 75

FIG. 6. It is formed of an elongated rhenium wire arranged in a planar bifilar spiral configuration. A lead 81 (FIG. 5) is coupled to the cathode 63 and to the grid 64 for establishing a same DC potential at these electrodes. The first anode 56 (FIG. 8) is formed of two relatively thin plates of ferromagnetic material. One of the plates includes a field forming hollow spherically-shaped integral segment 82 of diameter D_2 and radius R2, extending toward and into the grid electrode 64. An aperture 68 is formed in this segment. The relative physical dimensions of the grid 64 and segment 82 are given by $R_1 > h_1$ and $R_1 > R_2$.

The cathode electrode arrangement is particularly ad-

vantageous in that a relatively large electron emission surface area is provided. The bifilar spiral arrangement causes the 15 center of the cathode configuration to operate at a relatively higher temperature and to operate as the cathode segment of highest current emission along the elongated cathode. Because of this configuration, portions on the cathode further 20 away from the center run relatively cooler and have less emis-

sion. This simulates an ideal arrangement for focusing in which the cathode electrode is physically positioned on a lower inner surface of the spherically-shaped grid electrode. Accordingly, electron beam densities for enhancing the efficiency of the ion source are provided.

The configuration of the grid electrode 64, and the field focusing electrode 82 define electrostatic fields adapted for focusing the relatively large surface of the cathode electrode at the aperture 68. In establishing this focusing field the grid electrode 64 creates an equipotential surface and the field lines extend approximately radially toward the center of the sphere. Electrons emerging from the volume contained by grid electrode 64 are thereby directed conically to the aperture 68.

Thus, a relatively large current density is provided and is effectively focused in a volume of relatively small dimensions with a noncomplex electrode structure.

In accordance with another feature of the invention, the lines of magnetic flux 84 (FIG. 5) which extend in an axial direction through the ionization chamber are diverted from the cathode source of electrons and from the grid 64 by the first anode 56 formed by the two members of ferromagnetic material and by the pole piece 54. The desired focusing action in the vicinity of the cathode and grid electrodes thereby remains substantially undisturbed by the magnetic field.

In a particular configuration given by way of example and not deemed limiting in any respect, we have found the following ion source to exhibit a relatively increased efficiency:

Dimensions of the ionization chamber as illustrated in FIG. 50 9 are:

W = 0.310'' L = 0.210''H = 0.400''

Pole pieces 54 and 52 formed of ferromagnetic material having the dimensions as illustrated in FIG. 10 are:

- a=.230'
- b=.625''
- c = .350''
- d=.1575''
- e = .240''

$$f = .290^{11}$$

 $g = .310^{11}$

Pole pieces 48 and 50 have the same dimensions as corresponding pole pieces 54 and 52.

Cathode electrode 78 formed of rhenium and presenting an in.².

 $R_1 = .100''$

$$R_2 = .050''$$

Ε

 $E_2 = 1925 V DC$

 $E_3 = 2040 V DC$

$$h_1 = .030''$$

k = .015''

Aperture $68 = .050^{\prime\prime}$ diameter

m=.030''

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toward said grid electrode.

Thus an ion source of relatively high efficiency has been described wherein the electron beam density is substantially increased and is readily focused at a first anode electrode aperture with a relatively noncomplex electrode structure.

While we have illustrated and described a particular embodiment of our invention, it will be understood that various modifications may be made therein without departing from the spirit of the invention and the scope of the appended claims.

We claim:

1. An ion source for a mass spectrometer comprising:

- means, including an apertured first anode electrode having a spherically-shaped field focusing segment, defining an ionization chamber;
- a hollow grid electrode having a spherically-shaped segment 15 of radius R_1 and a frustum conically-shaped segment;
- a cathode electrode formed from an elongated wire and positioned within said grid electrode;
- an electron target electrode spaced apart from said cathode electrode and positioned for causing electrons emitted 20
- from said cathode to pass through said ionization chamber; and

means applying operating potentials to said electrodes.

2. The ion source of claim 1 wherein said first anode spherically-shaped field focusing segment has a radius R_2 where R_2 25 < R_1 and said grid electrode includes integral spherically- and frustum conically-shaped segments.

3. A high current intensity electron-optic arrangement comprising:

- a first anode electrode having a hollow spherically-shaped 30 field focusing segment of sphere radius R_2 ;
- a hollow grid electrode positioned relative to said first anode and having a spherically-shaped segment of radius R_1 and a frustum conically-shaped segment of height h_1 where $R_1 > h_1$; and
- a cathode electrode positioned within said grid electrode, said cathode formed from an elongated wire arranged in a planar configuration.

4. The electron-optic arrangement of claim 3 wherein said first anode field focusing segment extends and converges 40

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5. The electron-optic arrangement of claim 4 wherein $R_1 > R_2$.

6. The electron-optic arrangement of claim 4 wherein said first anode field focusing segment extends into said hollow grid electrode.

7. The electron-optic arrangement of claim 4 wherein said cathode electrode is formed as a bifilar spiral.

8. The electron-optic arrangement of claim 7 wherein said 10 bifilar spiral is formed substantially in one plane.

- 9. An ion source for a mass spectrometer comprising:
- means defining an ionization chamber, said means including first and second oppositely positioned planar wall members;
- said first wall member comprising a first anode electrode and having a centrally located spherically-shaped apertured field focusing segment of sphere radius R₂ extending outwardly from the chamber;
- said second wall member having a centrally located aperture aligned with said aperture in said first wall member;
- an electron target electrode positioned without said chamber adjacent the aperture of said second wall member;
- a grid electrode formed as a hollow body including an integral semispherically shaped segment of sphere radius R_1 where $R_2 < R_1$ and an integral frustum conically-shaped segment, said conical segment having a convergent aperture in a cross section thereof;
- means supporting said grid electrode adjacent said first anode focusing segment for providing extension of said segment into said conical grid segment; and
- a directly heated cathode electrode formed of an elongated wire arranged in a bifilar planar configuration supported within the hemispherical grid segment.
 10. The ion source of claim 9 wherein said ionization
- 10. The ion source of claim 9 wherein said ionization chamber includes wall members providing a source of magnetic potential for establishing a generally longitudinal magnetic field within said chamber and said first anode is formed of a ferromagnetic material.

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