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Matsuda

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[54] RAPID SCANNING DOUBLE FOCUSING MASS SPECTROGRAPH

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Related U.S. Application Data

[63] Continuation-in-part of Ser. No. 702,469, Feb. 1, 1968, abandoned.

[52] **U.S. Cl.** ..... **250/41.9 ME**

[51] **Int. Cl.** **H01j 39/34**

[58] **Field of Search** ...250/41.9 G, 41.9 SA, 41.9 ME

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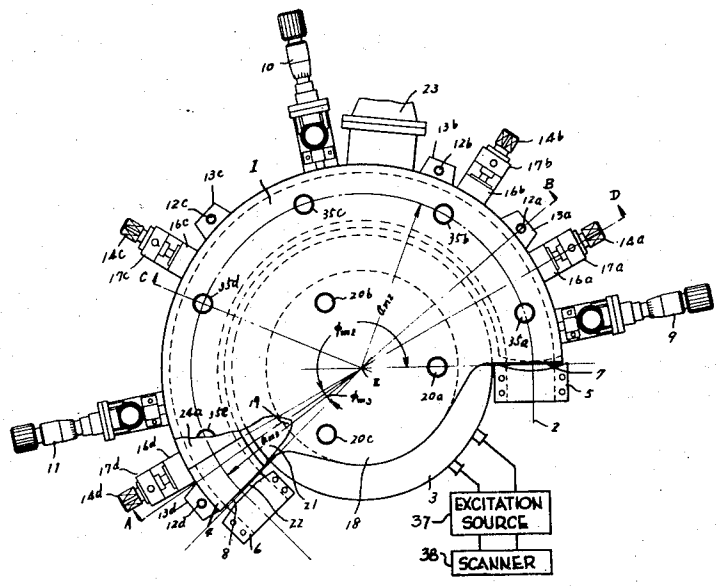
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[57] **ABSTRACT**

A rapid scanning double focusing mass spectrograph having a source of ions, means for producing a toroidal electric field, means for producing a homogeneous magnetic field and means for producing an r^{-1} magnetic field, wherein the r^{-1} magnetic field and the homogeneous magnetic field are excited simultaneously with a single energizing magnet and upper and lower parallel pole pieces of the means for producing a homogeneous magnetic field are secured to the rear ends of the upper and lower conical pole pieces of the means for producing an r^{-1} magnetic field with the curvature radii of the two means being kept equal.

6 Claims, 3 Drawing Figures



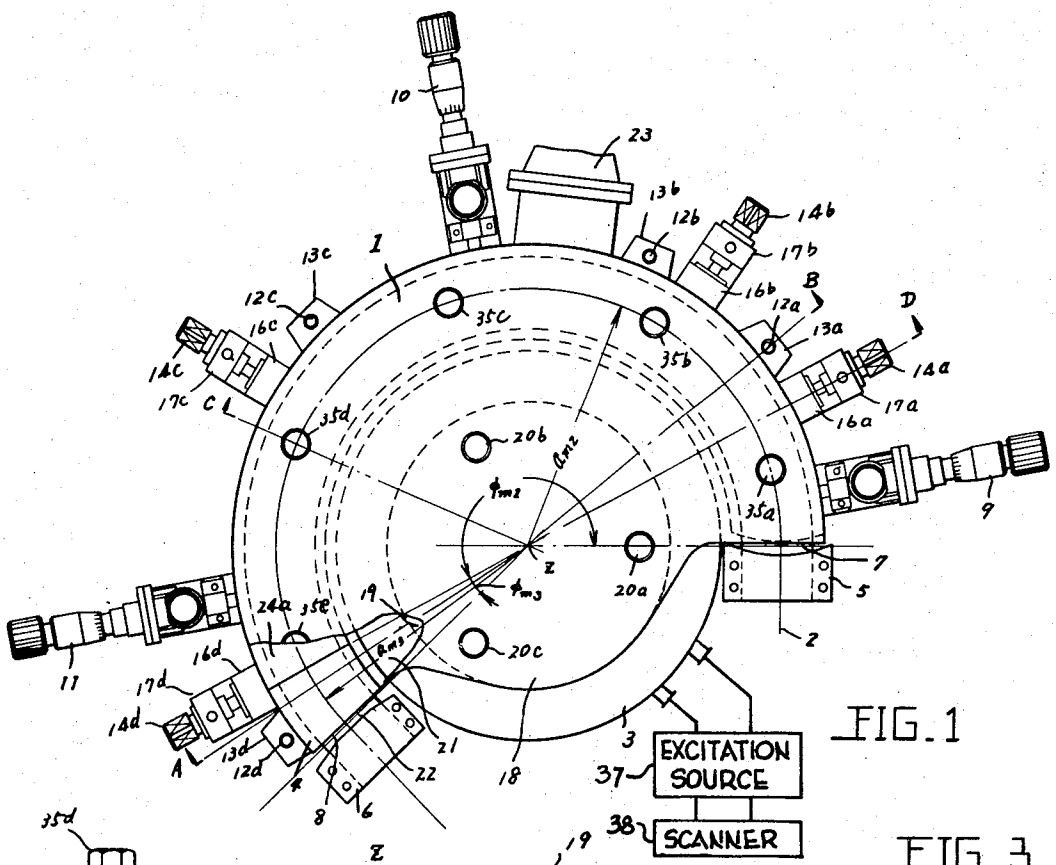


FIG. 1

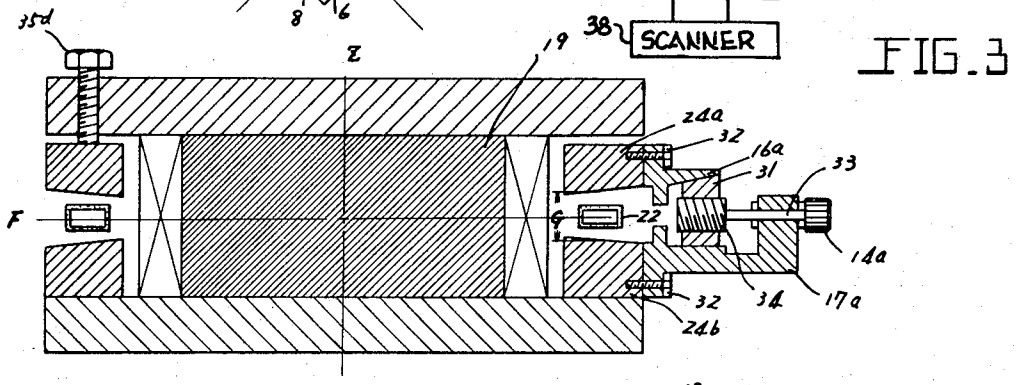


FIG. 3

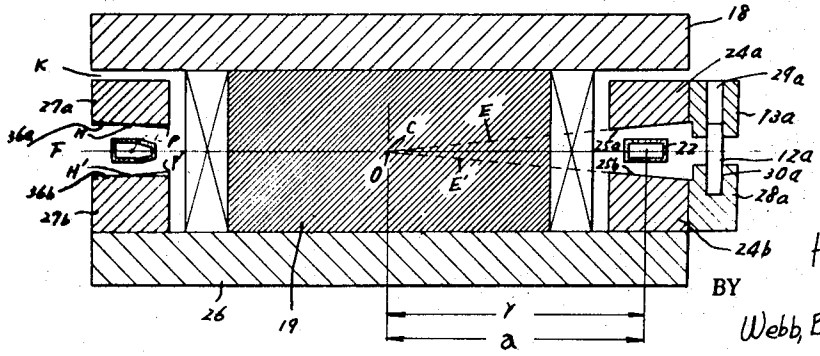


FIG. 2

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RAPID SCANNING DOUBLE FOCUSING MASS SPECTROGRAPH

This application is a continuation-in-part of application Ser. No. 702,469, filed Feb. 1, 1968, now abandoned.

My invention relates to rapid scanning mass spectrographs or spectrometers having large mass dispersion and high resolving power for the precise determination of atomic masses. In particular, my invention relates to double focusing mass spectrographs.

The requirements of the magnetic field or fields in a mass spectrometer are twofold: they must separate the ions in an ion beam according to their mass to charge (m/e) ratios. At the same time they must focus the paths of ions having the same m/e ratios but which enter the field along slightly different paths. In the simple mass spectrometer, a uniform magnetic field causes the greater dispersion of ions having different m/e ratios after the ions have been turned through 180°. This is also the point of maximum focusing for the paths of ions having the same m/e ratio. Beyond 180° the focus is lessened and the dispersion decreases. Hypothetically, at 360° the paths of all ions regardless of mass to charge ratio converge on the point at which they entered the field.

In a simple mass spectrometer that focuses the beam at the end of a 180° turn, the only manner in which dispersion can be increased, is to increase the radius of curvature of the ion path. To increase the radius of curvature of the ion path, it is necessary to use larger and larger pole pieces and cores which become awkward and unwieldy for the large dispersions desired for analysis of organic compounds.

Also, in a double focusing mass spectrograph, by enlarging the radius of the ion orbit, it is possible to obtain high resolutions. For technical reasons, it is very difficult to sufficiently enlarge the linear dimensions of the apparatus thereby making this method rather impractical.

Recently, a new type of mass spectrograph with a large mass dispersion that provides an ultra high resolution of several hundred thousands or more without requiring large linear dimensions has been developed. This has been accomplished by utilization of an r^{-1} magnetic field for the mass dispersing action. The r^{-1} magnetic field, which I first proposed, is characterized by a magnetic field strength in the Y-direction, i.e., in the radial direction, that decreases in accordance with the increase in the radius of the magnetic field, according to the formula:

$$B = B_o (r/a)^{-1},$$

wherein B_o = the magnetic field strength along the central orbit,

a = the curvature radius of the central ion beam in the magnetic field, and

r = the radial component distance of a given point. The r^{-1} magnetic field has no focusing action, but rather has a dispersive action in the radial direction, that is, in the direction of the plane determined by the central orbit. A detailed description of the apparatus appears in "A New Mass Spectrograph With Very Large Dispersion," published in Zeitschrift für Naturforschung, Vol. 21a, pages 25–33, 1966.

In a mass analyzing device of that kind, a sector type toroidal electric field having a deflection angle of 118.7° and a means radius of 30cm is used as the first focusing field to compensate for the large energy dispersion that is caused by the r^{-1} magnetic field. For the second focusing field, a homogeneous magnetic field of 120cm mean radius is used to focus ions having the same mass on a photographic plate or the like. A sector type r^{-1} magnetic field having a deflecting angle of 198.1° and a mean radius of 22cm, the strength of which varies in proportion to r^{-1}, is inserted between the toroidal electric field and the homogeneous magnetic field.

With this arrangement consisting of three fields, two focusing and one dispersing, the double focusing condition is given by

$$(2/Ke) \sin Ke\,\phi\, e = \sin \phi\, m_3 + \phi\, m_2,$$

where Ke is the electric field characterizing constant, and ϕe, ϕm_2 and ϕm_3 are the deflecting angles of the toroidal electric field, dispersing magnetic field and focusing magnetic field, respectively.

In order to reproduce an extremely high resolution mass spectrum, (M/Δ M; 500,000 or better), it is necessary to precisely adjust the electric currents of the exciting coils producing the two magnetic fields so as to prevent the ion beam from deviating from the original optimum path of travel. In instruments designed according to the prior art, the ion beam, after passing through the toroidal electric field, deviates from the optimum path of travel when dispersing magnetic field intensity is changed, since the dispersing magnet and focusing magnet coils are energized by different magnet power supplies. Extremely precise control of the focusing magnetic field intensity is necessary to restore the beam to its optimum path of travel in accordance with the change in the dispersing magnetic field intensity. Even when exercising the aforementioned care during control, it is very difficult to adjust the strength of the dispersing magnetic field so as to maintain the intensity ratio between the two magnetic fields constant at all times, because of the presence of residual electromagnetic hysteresis. Furthermore, the strengths of the two magnetic fields are required to be varied in proportion to each other and at the same time the intensity ratio between the magnetic fields must be maintained constant at all times with a stability of an order of more than 10^{-4}. It is impossible, however, to detect the intensity ratio between the two magnetic fields with an accuracy of more than 10^{-3} when using a Hall element.

As a result, the optimum operating condition of the mass spectrograph cannot be attained without precise and time consuming adjustments which includes the actual emittance of ions from the ion source to the detector for travel along the optimum ion orbit that originally provided the high resolution mass spectrum. Consequently, each time the power supplied to energize the dispersing magnet and focusing magnet is changed in accordance with the change in the measuring condition, it is necessary to repeat the entire procedure several times until the optimum operating condition of the two magnetic fields, both with respect to beam position and beam focusing, is attained.

Furthermore, due to the presence of the residual electromagnetic hysteresis, it is extremely difficult to

scan the mass spectrum of a sample which requires that the intensity ratio between the two magnetic fields be maintained constant at all times. Moreover, since the exciting current to the respective magnets must be adequately stabilized (10^{-7}), it is necessary to provide magnetic field exciting current stabilizers, which are not only very expensive, but tend to make the entire apparatus very large and bulky.

My invention provides an improved mass spectrograph that overcomes the aforementioned disadvantages, so that a resolving power in the order of from 100,000–300,000 over the entire range is ensured by making adjustment at only one point of the mass to charge ratio of the charged particles.

In practice, the intensities of the r^{-1} magnetic field and homogenious magnetic field cannot be scanned at high speed simultaneously and yet, with this invention, it is possible. Moreover, since the above described high resolving power can be obtained over a very wide range with this high speed scanning, the mass to charge ratio of each spectrum can be measured accurately.

Other novel features and advantages will become apparent by reading the following detailed specification in conjunction with the accompanying drawings in which:

FIG. 1 is a plan view, partly broken away, of the focusing and dispersing means of my invention;

FIG. 2 is a cross-section of the invention taken on line A–Z–B of FIG. 1; and,

FIG. 3 is a cross-section of my invention taken on line C–Z–D of FIG. 1.

Briefly according to applicant's invention, a r^{-1} magnetic field and a homogeneous magnetic field are placed about the same reasonably sized magnetic core. The two magnetic fields having the same curvature radius are energized with a single magnet. The magnetic core is energized through a scanner and an excitation source. Dispersion is not determined by the radius of curvature of the ion path. It is controlled by the angle through which the ion beam is turned. The r^{-1} field does not focus the ion beams at the end of a 180° turn. Ions of different m/e ratios emerge from the r^{-1} magnetic field at different angles; however, all ions having the same m/e are traveling in parallel paths. The homogeneous field following the r^{-1} magnetic field focuses the ions emerging from the r^{-1} field. The focal length of this field and the dispersing angle of the r^{-1} field control the resolving power of applicant's spectrometer. Hence, the combination of the r^{-1} magnetic field with the homogeneous magnetic field results in a spectrometer that does not have to be focused at the end of a 180° turn and which is not dependent upon the increasing radius of curvature to obtain increased resolution. Large pole pieces are not required. The r^{-1} magnetic field and the homogeneous magnetic fields are placed around the same reasonable sized core enabling the fields to be rapidly swept simultaneously over a range of intensities such that the intensity ratio between both fields is maintained constant. In this way, a high resolving power over the entire scanning range is achieved.

Referring to FIG. 1, and r^{-1} magnetic field 1 and a central ion beam path 2 having a curvature radius am_2, for example 30cm, are dependent upon the size of energizing magnet 3. The r^{-1} magnetic field has a focusing effect on the ion beam in the axial direction, that is, in the direction vertical to the plane determined by the orbit. In order to obtain optimum mass dispersion, it is preferable to make the deflecting angle $\phi\ m_2$ as large as possible. For example, taking into consideration the focusing effect, an angle of 200.32° could be used where am_2=30cm. Though not shown in FIG. 1, a toroidal electric field is arranged as the first focusing field. The deflection angle of the toroidal electric field having 30cm curvature radius is 112.15°. A homogeneous magnetic field 4 serves as the second focusing field. The curvature radius am_3 of the ion beam, in this case, is also 30cm to permit the focusing magnetic field to be placed immediately after the dispersing magnetic field. The deflecting angle $\phi\ m_3$ of the focusing magnetic field is 13.59° which is determined by the double focusing condition. From the double focusing condition it is apparent that curvature radii of the ion beam in the three fields, the distance between the toroidal electric field and the dispersing magnetic field, and the distance between the dispersing magnetic field and the focusing magnetic field are independent of the double focusing condition. Herzog shunts 5 and 6 (an explanation of the mechanisms has been omitted for the sake of clarity) are arranged at entrance 7 of the dispersing field and at exit 8 of the focusing magnetic field to reduce stray magnetic fields as much as possible and for the realization of a good focusing condition. Beam current monitors 9, 10 and 11 (an explanation of the mechanisms has also been omitted for the sake of clarity) are arranged at points along the ion path for monitoring purposes. Knock pins 12*a*12*b*, 12*c* and 12*d* made of nonmagnetic material are arranged in holes in the upper cylindrical supports 13*a*, 13*b*, 13*c* and 13*d* so as to be in sliding contact with the upper cylindrical supports. Rotary knobs 14*a*, 14*b*, 14*c* and 14*d* are mounted on the exterior of an upper conical pole piece 24*a* and a lower conical pole piece (not shown) so as to impart forward and backward movement to four distant wedge-shaped pieces of phosphor bronze 31, one of which is shown in FIG. 3, arranged between the respective upper collars 16*a*, 16*b*, 16*c* and 16*d* and lower collars 17*a*, 17*b*, 17*c* and 17*d*. The upper yoke plate 18 is secured to the upper surface of the centrally arranged core 19 of magnetic material by means of three screws 20*a*, 20*b* and 20*c*. Though not shown, the lower yoke plate is also secured to the lower surface of the core 19 in a like manner. By means of this arrangement, the upper and lower yoke plates are electromagnetically connected to the core, on which an exciting coil 21 is accommodated. Z indicates the vertical axis as the center of the two magnetic fields. An analyzer tube 22 made of nonmagnetic material is housed in a gap formed in the peripheries of the conical and parallel pole pieces. The interior of the tube is maintained at a high vacuum by means of a vacuum pump (not shown), connected to the tube via a conduit 23.

To enable the r^{-1} magnetic field to be generated exactly in the median plane, that is, in the plane determined by the central orbit, the upper and lower conical pole pieces 24*a* and 24*b*, made of soft iron, are symmetrically arranged with respect to the plane of symmetry F as shown in FIG. 2.

In addition a 15mm gap G at the mean radius is provided between the confronting conical pole faces 25*a*

and **25***b* and the conical pole faces are shaped so as to converge at point C that coincides with the intersection point O of the plane F of symmetry and the axis Z which is vertical to the plane. In other words, the confronting conical pole faces **25***a* and **25***b* are disposed so that the distance r between the path of the central ion beam and the intersection point C of the converging extension lines E–E′ is equal to the curvature radius a of the ion beam in the dispersing magnetic field.

However, the nonuniformity of gap G between the confronting conical pole pieces and variations in the magnetic characteristics of the magnetic conical pole pieces introduces asymmetries in the r^{-1} magnetic field that have a disturbing effect on the path of the ion beam entering or leaving the dispersing magnetic field. Though great care is usually employed in selecting and machining the pole piece assembly, additional correction is necessary because of the nonuniformity and variations to ensure that the dispersing magnetic field is distributed exactly in proportion to r^{-1} at all points around the ion beam path, thus ensuring a high resolution mass spectrum.

The gap between the parallel pole faces of the focusing magnetic field is not mechanically controllable and, therefore, the strength of the focusing magnetic field necessarily has to be controlled by varying the exciting current of the energizing magnet to maintain the ion beam in the calculated ion path.

In order to overcome these disadvantages, the present invention provides an arrangement whereby the parallel pole pieces are welded to the rear ends of the conical pole pieces. As shown in FIG. **2**, the top and bottom of the centrally arranged core **19** made of ferromagnetic material, abuts the upper and lower yoke plates **18** and **26**. On the lower yoke plate the lower conical pole piece **24***b* is rigidly mounted. The upper and lower parallel pole pieces **27***a* and **27***b* with confronting upper and lower parallel pole faces **36***a* and **36***b* are intercrossing with the confronting upper and lower conical pole faces **25***a* and **25***b* in points H and H′ which are located directly above and below path **2** of the central ion beam on the rear ends P and P′ of the upper and lower conical pole pieces **24***a* and **24***b*, so that the curvature radius of the ion beam in the focusing magnetic field can be maintained equal to that of the ion beam in the dispersing magnetic field.

In the operation of the mass spectrograph, according to the present invention, it is also necessary to adjust pole gap G in the first place so that the r^{-1} magnetic field is distributed exactly in proportion to r^{-1} at all points around the ion beam path. Towards this end, an air gap K, for example, 0.5mm, is provided between the lower surface of the upper yoke plate and the upper surfaces of the upper conical and parallel pole pieces. Knock pin **12***a* is slidably arranged in holes **29***a* and **30***a* in the upper and lower cylindrical supports **13***a* and **28***a* which are in turn fixed to the respective outside walls of the upper and lower conical pole pieces. Three other knock pins are also arranged in respective holes in the cylindrical supports in like manner.

As shown in FIGS. **1** and **3**, distant wedge-shaped piece **31** is arranged between the upper and lower collars **16***a* and **17***a* so as to be in sliding contact with the said collars. The upper and lower collars **16***a* and **17***a* are secured to the outside walls of the upper and lower conical pole pieces **24***a* and **24***b* by means of screws **32**. Control knob **14***a* is connected to a shaft **33** in torque transfer relation, the inner end of the shaft is provided with a screw thread **34** which is rotatable within the distant wedge-shaped piece **31**. The screw thread, when rotated by means of the control knob **14***a*, imparts an upward and downward movement to upper collar **16***a* and hence to the upper conical pole piece **24***a*. At the same time other wedge-shaped pieces are also controlled in a like manner by the respective control knobs **14***b*, **14***c* and **14***d*, thus moving the upper conical pole piece **24***a* up and down, i.e., parallel to the Z axis which is vertical to the plane F of symmetry. Once the optimum position of the upper conical pole piece is obtained, bolt **35***d* and the other bolts **35***a*, **35***b*, **35***c* and **35***e* are also turned to press the upper conical pole piece and, hence, the upper collars onto the distant wedge-shaped pieces. This operation adjusts the dispersing magnetic field to be changed exactly in proportion to r^{-1} in the plane of symmetry F in which the path of the central ion beam lies. Although the gap between the parallel pole pieces is simultaneously changed, the curvature radius of the ion beam in the focusing magnetic field is maintained equal to that of the ion beam in the dispersing magnetic field at all times, since the upper parallel pole face **36***a* which is intercrossing with the upper conical pole face **25***a* in a point directly above the path of the central ion beam is moved up and down, i.e., parallel to the vertical axis Z together with the upper conical pole face. Due to the common energizing magnet **3**, the strength of the focusing magnetic field along the ion beam path is kept equal to that of the dispersing magnetic field along the ion beam path at all times, and the adverse effect of the fringing magnetic fields on the ion beam path is reduced to negligible proportions.

Thus, in the mass spectrograph with large mass dispersion constructed according to the present invention, the dispersing magnetic field strength along the ion beam path is kept equal to the dispersing magnetic field strength along the ion beam path by generating the two magnetic fields with a single energizing magnet **3** exactly by an excitation source **37**. The time required to control the strength of the focusing magnetic field so that it varies in proportion to the strength of the dispersing magnetic field is reduced to a great extent. Rapid scanning of the mass spectrum of a sample which requires the strengths of the two magnetic fields to be varied and at the same time the intensity ratio between the two magnetic fields to be kept constant at all times, has been made possible by connecting a scanner **38** to the excitation source **37**.

While I have shown and described a preferred embodiment of my invention, it may otherwise be embodied within the scope of the appended claims.

I claim:

1. In a rapid scanning double focusing mass spectrograph having an ion source, two focusing means and a dispersing means wherein an ion beam passes through a first focusing means to a dispersing means and then through a second focusing means prior to impingement upon a detector, the improvement comprising:

A. a means for producing a dispersing field and a second focusing field in which the curvature of radii are equal; said dispersing field being an r^{-1}

magnetic field and said second focusing field being an homogeneous magnetic field; and,

B. a means comprising a common electromagnetic core for exciting both fields simultaneously over a range of intensities such that the intensity ratio between both fields is maintained constant.

2. The improvement set forth in claim 1 wherein the means for producing the dispersing field, r^{-1} magnetic field, comprise upper and lower confronting conical pole pieces symmetrically arranged about the ion beam orbit plane, said pole pieces having projection lines that intersect on the orbit plane and vertical axis of the r^{-1} and homogeneous magnetic fields, the centers of said pole pieces lying above and below the central ion beam.

3. The improvement set forth in claim 2 wherein said means for producing the second focusing field, homogeneous magnetic field, comprise upper and lower parallel pole pieces, the centers of said pole pieces lying above and below the central ion beam.

4. The improvement set forth in claim 3 wherein the upper pole pieces are joined together and the lower pole pieces are joined together.

5. The improvement set forth in claim 3 wherein the gap between the upper and lower pole pieces is adapted to vertical adjustment by adjusting means.

6. The improvement set forth in claim 5 wherein said adjusting means comprise upper and lower collars mounted on the upper and lower conical pole pieces, nonmagnetic wedge-shaped elements slidably mounted between said collars, and means for moving said wedge-shaped elements inwardly and outwardly from said pole pieces to vertically adjust said pole pieces.

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