

[54] **DOUBLE PASS COAXIAL CYLINDER
ANALYZER WITH RETARDING
SPHERICAL GRIDS**

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250/41.9 Me

[51] Int. Cl. **H01j 37/26**

[58] Field of Search. 250/41.9 ME, 49.5 PE, 49.5 AE

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Assistant Examiner—Harold A. Dixon

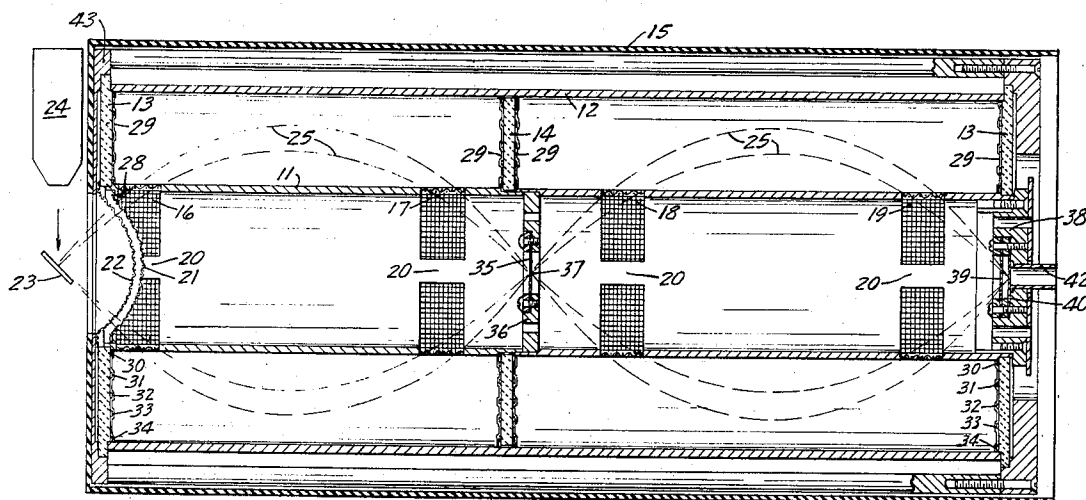
Attorney—Schroeder, Siegfried, Ryan & Vidas

[57]

ABSTRACT

A charged particle analyzer is provided wherein a retarding grid consisting of two spaced screen members formed of spherical sections having a concentric center located at the surface of the sample being analyzed is utilized to control the entering velocity of the charged particles into a double pass, coaxial cylinder analyzer. In a specific embodiment, the invention is utilized in an electron spectroscopy for chemical analysis (ESCA) apparatus.

7 Claims, 6 Drawing Figures



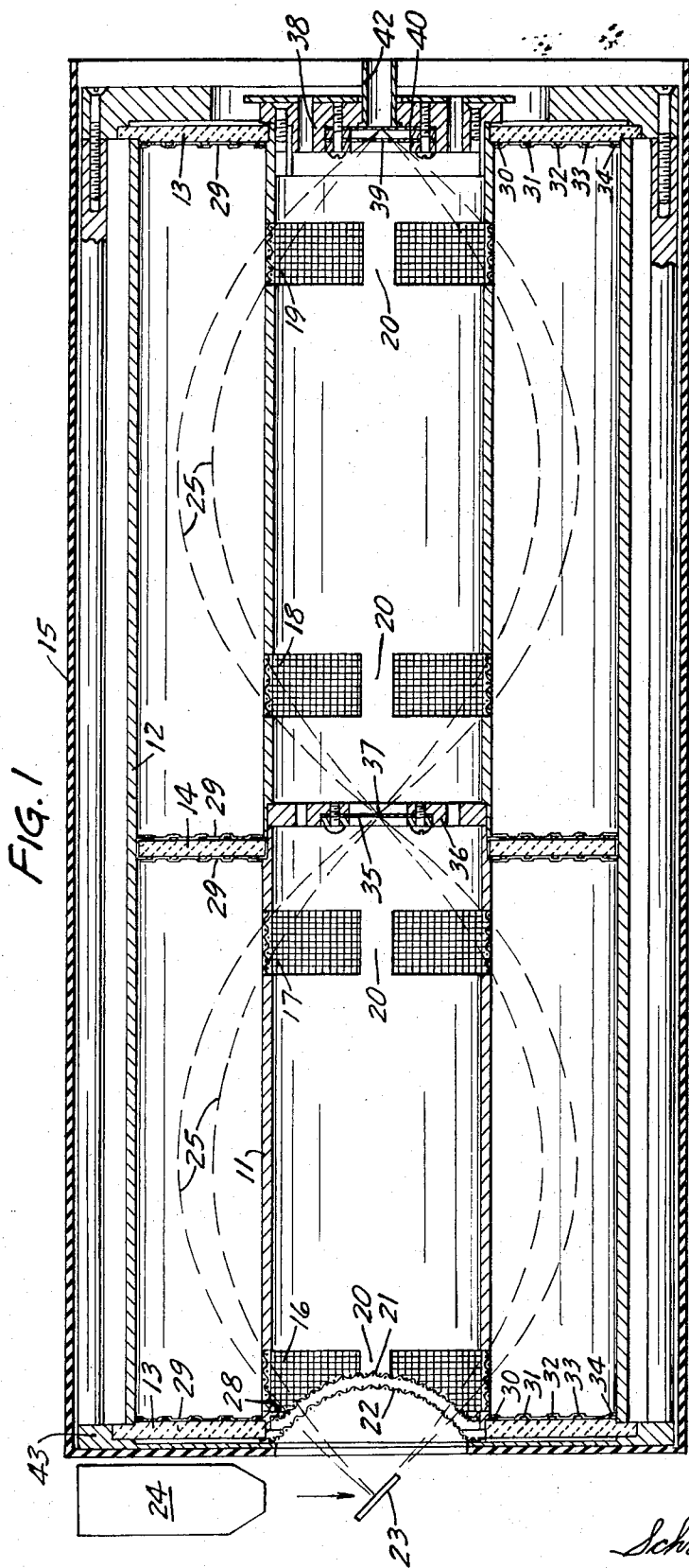


FIG. 3

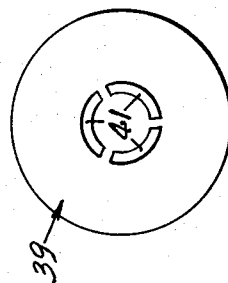
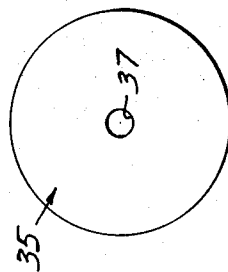


FIG. 2



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FIG. 4

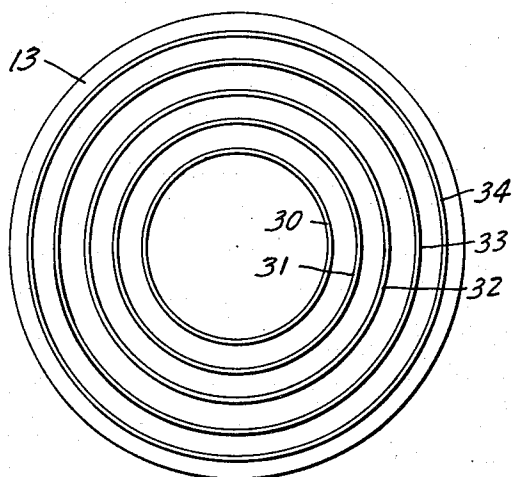


FIG. 5

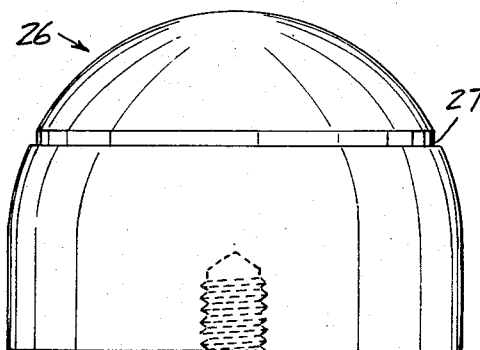
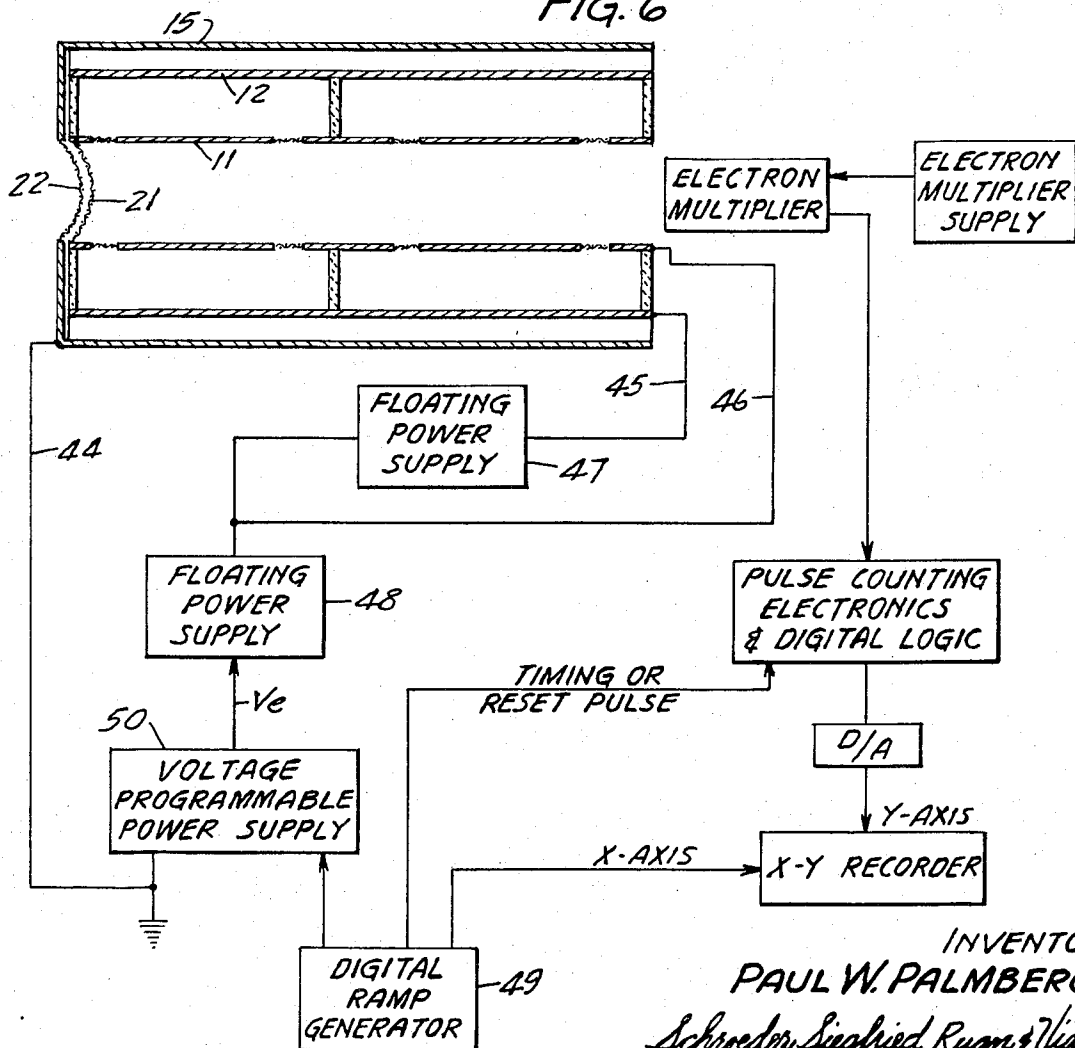


FIG. 6



DOUBLE PASS COAXIAL CYLINDER ANALYZER WITH RETARDING SPHERICAL GRIDS

The present invention is directed to apparatus useful in analyzing the energy distribution of charged particles being emitted from a test sample after being bombarded with some form of high energy radiation such as protons, electrons, or X-ray. While an analyzer in accordance with the invention is anticipated to find a wide variety of use, it will be described with particularity in its use in the field of electron spectroscopy for chemical analysis (ESCA).

In ESCA type analyses, a sample to be analyzed is irradiated with generally monochromatic soft X-rays. These may be Al K α X-rays which have an energy of about 1486 eV. Other sources of activation may be used, such as an electron gun or high energy photons. The sample, in turn, gives off emitted electrons which have an energy range determined by the elements that the sample is made of. When the energy distribution is determined with sufficiently high resolution, one can also establish the chemical environment which a particular atom has based upon the energy distribution of the emitted electrons.

It is toward the production of an apparatus having an improved degree of resolution and sensitivity that the present invention is directed. The objects of the invention are accomplished through a combination of a specially constructed retarding grid and a double pass cylindrical mirror analyzer.

IN THE DRAWINGS

FIG. 1 is a side elevational view, mostly in cross-section, of an analyzer and certain peripheral equipment in accordance with the invention;

FIG. 2 is a front elevational view of an aperture disc for use in the apparatus of FIG. 1;

FIG. 3 is a front elevational view of a second aperture for use in the apparatus of FIG. 1;

FIG. 4 is a front elevational view of a field termination plate utilized in the apparatus of FIG. 1;

FIG. 5 is a side elevational view of a grid forming mold; and

FIG. 6 is a side cross-sectional view of the cylinder analyzer of FIG. 1 with a schematic illustration of circuitry for use therewith.

The resolution of a cylinder analyzer is determined by the geometry of the instrument. This means that $(\Delta E)/E$ is equal to a constant determined by the geometry of the instrument. If one reduces the energy of electrons entering the cylinder analyzer, one can increase the resolution of the instrument. For example, if the instrument has a designed resolution of 0.3 percent, this means that for electrons having an initial energy of 1000 eV, the resolution will be 3 eV. By decreasing the energy of electrons from an initial energy of 1000 eV to 100 eV, with a retarding grid in accordance with the invention, one can increase the resolution of the measurement from 3 eV to 0.3 eV. This resolution proves adequate for ESCA measurements. I have found, however, that it is critical that the field utilized to retard the electrons being analyzed be one of high uniformity and radially directed to the source of electrons. To provide this highly uniform field, the retarding grid assembly in accordance with my invention is constructed of two concentric spherical sections, as will be described hereinbelow.

Ideally, an analysis should be performed on electrons being emitted from a point source on the sample being analyzed. If this ideal was actually the case, the cylinder analyzer would perform in an optimum manner, as focusing of the electrons analyzed would be less of a problem. However, in virtually every form of analysis utilizing a cylinder analyzer, this ideal condition is not met. In particular, when one utilized X-rays as the irradiating source, it is extremely difficult to concentrate the X-rays into a region that could be considered a point source. Imaging of the source of the electrons in the sample then becomes a problem, and the resolution of the instrument is decreased. By utilizing a retarding grid construction in accordance with my invention in conjunction with a double pass analyzer, as will be described in greater detail below, my invention accomplishes its purposes of providing high resolution even though it uses a relatively large source. Through use of my invention, one can irradiate a large region of the sample without adversely affecting the analysis, as only those X-ray excited electrons within a well-defined region are able to pass through both regions of the analyzer. The double grid increases the virtual source size by deflecting electrons which do not originate from the ideal point source. Those electrons which are deflected during passage through the grids will not have proper trajectories to pass through both analyzers.

Turning first to FIG. 1, there is illustrated in primarily cross-sectional view an analyzer in accordance with the present invention. The apparatus in accordance with FIG. 1 includes an inner cylinder 11 and an outer cylinder 12, which are held in concentric alignment with one another by end ceramic plates 13 and a center ceramic plate 14. Cylinders 11 and 12 are constructed of a non-magnetic metal like copper, as are other metallic portions unless noted otherwise. Surrounding substantially the entire construction is a magnetic shielding member 15 which may be mu metal.

Inner cylinder 11 is provided with a series of annular openings 16, 17, 18 and 19 around the periphery thereof. Small regions 20 of the original cylinder wall are retained to support sections of the cylinder intermediate openings 16, 17, 18 and 19. Openings 16, 17, 18 and 19 are desirably covered by a fine mesh metallic screen which is desirably of about 100 lines per inch and has a transparency of about 80 percent. This screen permits the majority of the electrons or other charged particles being analyzed to pass through the screen while substantially eliminating electric field fringing due to the discontinuity of the walls produced by these openings.

One end of the inner cylinder 11 is partially closed by grids 21 and 22, which will be described in greater detail below. These grids are sphere segments having a common center of symmetry. At the center of symmetry is located the sample 23 (shown schematically) which is to be analyzed by the instrument. That portion of the sample to be analyzed is located at the center of symmetry of the grid members 21 and 22, and also on line with the axis of cylinders 11 and 12. A source of irradiation energy, such as an X-ray source, shown schematically as 24, is positioned to direct X-rays to strike as closely as possible to the symmetry point on the sample. The irradiation gives rise to emission of electrons from the surface layers of the sample, whose energies are functions of the composition of the material and of

the chemical environment in which the elements are associated. Some of the electrons emitted will follow the curved path illustrated by the dotted line 25 through the analyzer.

Turning to FIG. 5, there is illustrated a form for shaping of the grid members 21 and 22. Grid members 21 and 22 are formed of fine wire screen of the same type utilized in covering the openings of holes 16, 17, 18 and 19. The fine stainless steel screen is shaped over a block generally designated 26 of a material such as Teflon. The block will have a radius of curvature corresponding to that desired for the individual grid. In the instance of grid 21, the radius of curvature will, of course, be larger than that of grid 22. As typical dimensions, grid 21 will have a radius of about 1 inch, while grid 22 will have a radius of about 0.9 inches. These are suitable dimensional configurations for a cylinder analyzer wherein the inner tube has a diameter of about 2 inches, and the outer cylinder has an inner diameter of about 4 $\frac{3}{4}$ inches. For such a system, the overall length would be approximately 11 inches.

In the forming of the screens 21 and 22, a shoulder 27 is provided on the forming block so as to produce a flanged portion on the screen. Suitable die means are provided for pressing the screen member onto block 26 and shoulder 27 to produce the final configuration.

Referring again to FIG. 1, it will be seen that grid 22 is fastened by means such as welding to a metal plate 43 which is in electrical contact with outer magnetic shielding member 15. Shielding member 15 is electrically insulated from the balance of the assembly by ceramic 13. The inner grid 21 is in electrical contact with and mounted to a flange 28 projecting into the center of cylinder 11. Grid 21 and grid 22 are maintained in electrical isolation from one another by means of ceramic disc 13.

Spacing elements 13 and 14 are provided to maintain cylinders 11 and 12 in fixed relationship to one another and to provide a special field termination means to prevent field fringing. Each of these discs 13 and 14 is desirably formed of some electrically insulating material, which is preferably ceramic. Alumina or quartz are particularly suitable for the purpose. These discs are provided on the inner surface thereof with a conductive coating 29, having a high resistivity of about 30 megohms. This high resistance coating aids in the reduction of field fringing, which adversely effects the paths of the electrons as they pass through the analyzer.

As further aid to reducing the field fringing within the device, I construct the ceramic discs 13 and 14 in such a way that the surfaces interior to the analyzer have a plurality of concentric relatively high conducting annular rings, as is best seen in FIG. 4. In FIG. 4 there is generally illustrated a ceramic disc 13 in front elevational view, which has been provided with a series of concentric conductors in annular form and identified 30 through 34, respectively. These rings are concentric with one another and with the axis of the ceramic plate 13. Rings 30 through 34 are desirably formed by metallizing the surface of the ceramic discs 13 or 14 and then, by photolithographic masking and etching, removing the intermediate metal between the rings. The rings will desirably be about 0.005 inch in width and a few microns in thickness. Suitable metals include gold-chromium alloys that have been vacuum

sputtered onto the surface of the ceramic. The function of these rings is to provide equi-potential regions on the discs so as to minimize the effects caused by regions of resistivity that are not uniform within film 29.

As an alternative to the construction shown in FIG. 1, the spacer 13 at the sample end may be in the form of a truncated cone rather than a flat washer shape as shown. The forward edge of cylinder 12 would then be offset away from the sample, thus allowing a more simple positioning for the activation sources 24. These features are described in greater detail in my copending application filed of even date herewith entitled FIELD TERMINATION PLATE.

It should be noted with regard to FIG. 1 that the intermediate ceramic disc 14 has been treated on opposite surfaces thereof in a manner analogous to that described with regard to FIG. 4. It should also be appreciated that the conductive coating 29 is in electrical contact with cylinders 11 and 12 at the inner and outer extremities thereof. This can be readily achieved by metallizing the inner and outer edges of the ceramic discs 13 and 14.

At a point midway between the ends of inner cylinder 11, I provide an intermediate aperture disc 35. The aperture disc 35 is secured in fixed relationship to cylinder 11 by a mounting means generally indicated as 36. Disc 35 is shown in front elevational view in FIG. 2 and consists of a thin metallic disc (desirably of molybdenum) of about 0.003 inches thickness, having an opening 37 through the center thereof of about 0.036 inches diameter. This narrow aperture 37 acts as a filter to provide essentially a near point source of electrons for the second stage of the analyzer.

At the opposite end of the analyzer from sample 23 is a second set of aperture members mounted within a suitable mounting means generally identified 38. The end apertures 39 and 40 correspond to the configurations shown in FIGS. 3 and 2, respectively. Disc 40 is substantially identical to that of FIG. 2, both in thickness and dimension, while disc 39 is, as shown in FIG. 3, formed of a thin molybdenum disc of about 0.003 inches thickness, having a plurality of annular ring segment openings 41 that are positioned concentric with, but off of, the central axis of the disc. The function of such an off-axis aperture in disc 39 is described in the copending application of Bohn et al., Ser. No. 68,983, for AUGER ELECTRON SPECTROSCOPY.

As a final termination, there is provided a tubular member 42, which functions in the invention as the first dynode of a photomultiplier.

It will now be apparent to the reader that the interaction of the construction in accordance with the invention will provide an instrument capable of precise determination of the energy distribution of X-ray excited electrons over a wide energy range. The spherical grids 21 and 22, due to their configuration and positioning, act to controllably decrease the energy of the electrons passing therethrough so as to provide a resolution of measurement down into a range necessary for determining the atomic number and chemical state of the atom from which the electron originated. By use of the double pass cylindrical tube analyzer in accordance with the invention, the source of electrons is redefined as the electrons pass through the inter-

mediate aperture 37. The second stage of the double pass analyzer has, by virtue of the aperture 37, a "source" at a precisely known distance from the detector 42. Thus the energies of the electrons passing through the second stage is precisely defined by the potential between inner and outer cylinders 11 and 12. The detector for the electrons is an electron multiplier, of which element 42 forms the first dynode. The double pass arrangement further provides a construction wherein magnetic effects are essentially eliminated by the interior position of the entire second stage of the double pass analyzer. The absence of magnetic interference further provides a higher degree of precision of the overall instrument.

Turning now to FIG. 6, there is illustrated in cross section the analyzer in accordance with FIG. 1, with the principal portions thereof being shown in essentially schematic form. Electrical connections are made as shown to the various portions of the analyzer. The outer mu metal shield 15 is connected by a lead 44 to ground and to one side of a voltage programmable power supply. The outer cylinder 12 is electrically connected by lead 45 to a first floating power supply 47, while inner cylinder 11 is connected by means of lead 46 to the output of a second floating power supply 48. It will be noted that grid 21 is at the same potential as inner cylinder 11, while grid 22 will be at the same potential as mu metal shield 15, which is at ground. With power supplies 47 and 48 joined to cylinders 11 and 12 as shown, it will be appreciated that the potential of cylinder 12 will be at a value equal to the sum of power supplies 47, 48 and 50 while the potential of cylinder 11 will be at the potential of power supplies 48 and 50 alone.

A digital ramp generator 49 supplies the timing logic both to the voltage programmable power supply 50 and to the pulse counting electronics and digital logic unit. It also supplies a signal to an X-Y recorder as schematically illustrated in the figure. As the receiving element for the charged particles (electrons) which pass through the detector, there is an electron multiplier, which is shown as having a power supply, with its output of the multiplier going to a pulse counting electronic and digital logic unit. A digital to analog converter is utilized to convert the signal which then passes to the X-Y recorder as the Y axis signal.

Various commercial units are available for each of the units of electronics utilized in FIG. 6. As illustrative of suitable units, the floating power supplies 47 and 48 may conveniently be Hewlett Packard Models 6209B. The voltage programmable power supply 50 may be Model OPS-2000 of the Kepco Company of Flushing, New York. A digital ramp generator 49 is conveniently a laboratory computer PDP8/E, and a D/A converter available from Digital Equipment Corporation of Maynard, Massachusetts. A conventional X-Y recorder can be utilized such as Model 7004B of the Hewlett Packard Company.

In operation, the system of FIG. 6 functions substantially as follows. Sample 23 is irradiated by suitable means so as to emit electrons. These electrons then pass through screens 22 and 21, which are maintained at ground and at some suitable elevated negative potential, respectively. A scanning potential is applied to grid 21 to slow down the electrons passing between the two

grids to a value such that the resolution of the instrument can detect the necessary differences in electron energy required for the ESCA measurement. The voltage V_p applied between the inner cylinder 11 and outer cylinder 12, where 12 is negative with respect to 11, is the magnitude of the voltage that determines the pass energy E_p of the double pass analyzer:

$$E_p = k V_p$$

where k is a constant and depends upon the diameters of the inner and outer cylinders of the analyzer. The k is fixed for a particular analyzer geometry. In order to scan the range of energies of the electrons being emitted by the sample, one changes the potential of inner cylinder 11 and thus grid 21 to suitably slow down the electrons. A constant differential between the two cylinders is maintained by means of a floating power supply 47.

Alternatively, one may hold the potential between grids 22 and 21 at a constant value and scan by changing the potential between cylinders 11 and 12. However, under such a condition the energy spread, ΔE , of the instrument changes and makes measurements more complex.

Floating power supplies 47 and 48 are chosen so that their ratios of voltage are such that the energy of the electrons incident on grids 21 and 22 and passed by the analyzer is equal to V_e , the voltage of the voltage programmable power supply. It should be appreciated that power supply 48 will be positive with respect to power supply 50 so that the scans can be made down to zero energy.

As the electrons emitted from sample 23 pass through the grid arrangement 22-21 and enter the analyzer via opening 16, they are deflected by the more negative potential of outer cylinder 12 and follow the path generally marked 25. In this first stage of the analyzer, the electrons are partially resolved and generally are focused on plate 35 containing the aperture 37. Aperture 37 functions to further resolve the electrons into a nearly point source of definite size and distance from the ultimate receiver 42. In the second stage of the analyzer, the electrons which pass through opening 37 are further resolved and analyzed by passage through openings 18 and back through 19 and are ultimately focused by means of the double aperture system 39 and 40. The resulting analysis has a high signal to noise ratio and permits far greater precision of measurements than has been known with instruments known heretofore.

I claim:

1. An analyzer for determining the energy distribution of charged particles being emitted from a source, comprising:

- a. first and second metallic cylinders, the first of said cylinders having an outer diameter less than that of the inner diameter of the second cylinder and being positioned interior to the second cylinder, each of said cylinders having a common axis;
- b. disc-shaped aperture means positioned interior to and intermediate the ends of said first cylinder in a plane generally perpendicular to said axis and dividing said analyzer into first and second stages, said disc having an opening therethrough at said axis, the opening defining the source of charged particles for said second stage;

- c. said first cylinder defining a plurality of annularly spaced openings near each end of each stage of said first cylinder;
 - d. a retarding grid arrangement including first and second spherical section shaped screens positioned adjacent a first end of said first cylinder, said screens being spaced from one another and having a common center of symmetry at the source of charged particles and along said axis, said screens being in electrical isolation from one another, the second of said screens being in electrical contact with said first cylinder;
 - e. means for applying an electrical potential between said first screen and said second screen and for maintaining a predetermined potential between said first and second cylinders; and
 - f. charged particle detecting means positioned at the opposite end of said first cylinder.
2. An analyzer in accordance with claim 1 wherein said cylinders are held in spaced relation by annular electrically insulating spacers positioned at each end of the cylinders and at a point midway along the length thereof.
3. An analyzer in accordance with claim 2 wherein the surfaces of the spacers facing the interior space intermediate the first and second cylinders are coated with a high resistance conductive material which is in electrical contact with each of said cylinders.
4. An analyzer in accordance with claim 3 wherein the conductive coating includes a plurality of annular relatively high conductivity rings concentric with said axis to provide a plurality of equi-potential regions in said conductive coating.
5. An analyzer in accordance with claim 1 wherein the screens forming the retarding grid are spaced about 0.1 inches apart.
6. An analyzer in accordance with claim 4 wherein the screens are formed from line wire of about 100

lines per inch and about 80 percent transparency.

7. An electron spectroscopy chemical analysis system comprising:

- a. first and second metallic cylinders, the first of said cylinders having an outer diameter less than that of the inner diameter of the second cylinder and being positioned interior to the second cylinder, each of said cylinders having a common axis;
- b. disc-shaped aperture means positioned interior to and intermediate the ends along the length of said first cylinder in a plane perpendicular to said axis and dividing said analyzer into first and second stages, said disc having an opening at said axis, the opening defining the source of charged particles for said second stage;
- c. said first cylinder defining a plurality of annularly spaced openings near each end of each stage of said first cylinder;
- d. a retarding grid arrangement including first and second spherical section shaped screens positioned adjacent the first end of said first cylinder, said screens being spaced from one another and having a common center of symmetry along said axis, said screens being in electrical isolation from one another, the second of said screens being in electrical contact with said first cylinder;
- e. means for positioning a sample to be analyzed so that a portion thereof is along said axis and at the center of symmetry of said screens;
- f. X-ray source means arranged to direct a beam of X-rays onto said sample to be analyzed;
- g. means for applying electrical potential between said first screen and said second screen and for maintaining constant predetermined potential between said first and second cylinders; and
- h. electron detecting means positioned at the opposite end of said first cylinder.

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UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,699,331 Dated October 17, 1972

Inventor(x) Paul W. Palmberg

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

On the title page, following the inventor's name
insert:

assignor to Physical Electronics
Industries, Inc., Edina, Minnesota,
a corporation of Minnesota

Signed and sealed this 15th day of May 1973.

(SEAL)
Attest:

EDWARD M. FLETCHER, JR.
Attesting Officer

ROBERT GOTTSCHALK
Commissioner of Patents