

[54] ESCA X-RAY SOURCE

[75] Inventor: Paul E. Larson, Acton, Mass.

[73] Assignee: GCA Corporation, Bedford, Mass.

[22] Filed: May 23, 1973

[21] Appl. No.: 363,141

[52] U.S. Cl. 250/305, 250/493, 250/503

[51] Int. Cl. H01j 37/00

[58] Field of Search 250/399, 493, 305; 313/55, 313/330, 74, 503

[56]

References Cited

UNITED STATES PATENTS

2,797,333	6/1957	Reiffel	250/493
3,174,036	3/1965	Alexeff	313/93
3,510,656	5/1970	Hood	250/493
3,602,686	8/1971	Lempert	250/399

3,617,741	11/1971	Siegbahn.....	250/305
3,681,600	8/1972	Rigden et al.....	250/305
3,699,331	10/1972	Palmberg.....	250/305
3,766,381	10/1973	Watson.....	250/305
3,787,692	1/1974	Anderson.....	250/305

Primary Examiner—Archie R. Borchelt

Assistant Examiner—B. C. Anderson

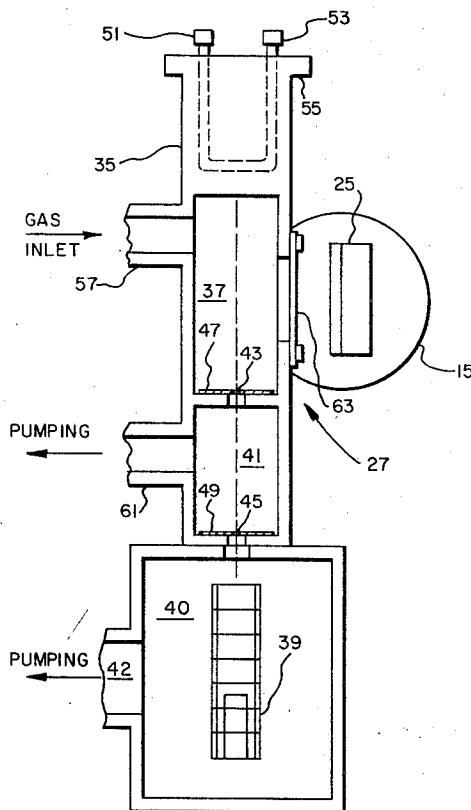
Attorney, Agent, or Firm—Kenway & Jenney

[57]

ABSTRACT

The X-ray source disclosed herein provides essentially monochromatic X-rays for sample irradiation in electron spectroscopic apparatus. The X-rays are generated by the impingement of a high energy electron beam on a gas under substantial pressure, pumping being provided to maintain a vacuum at the electron beam source in the presence of escaping target gas.

6 Claims, 2 Drawing Figures



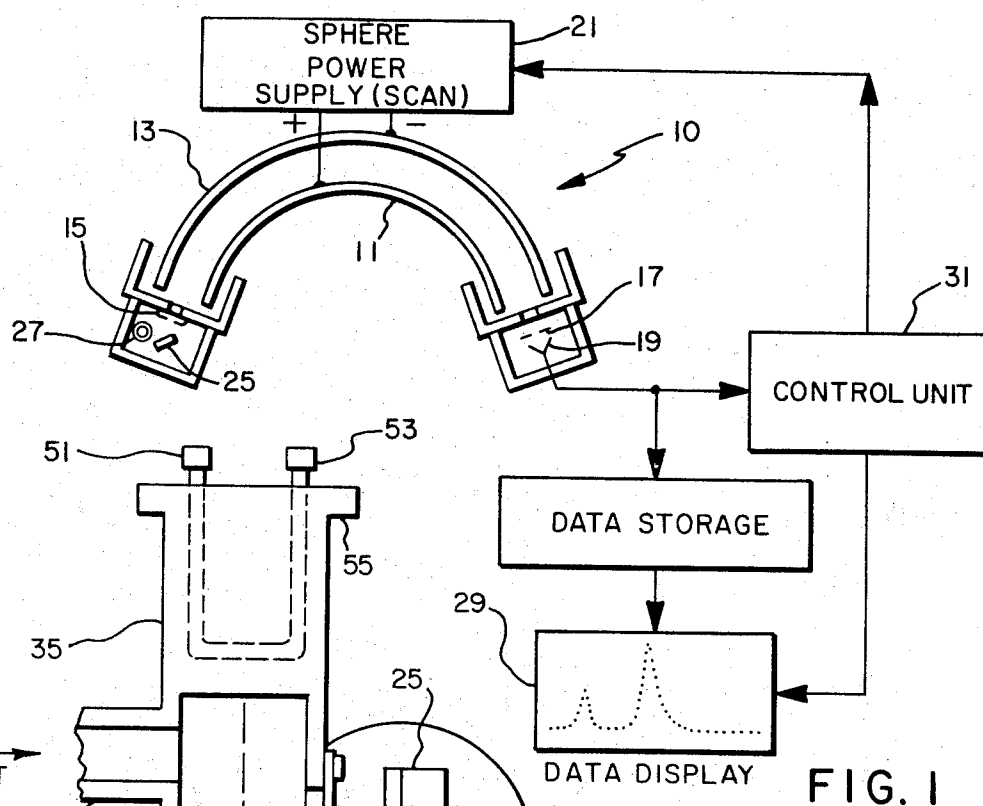


FIG. 1

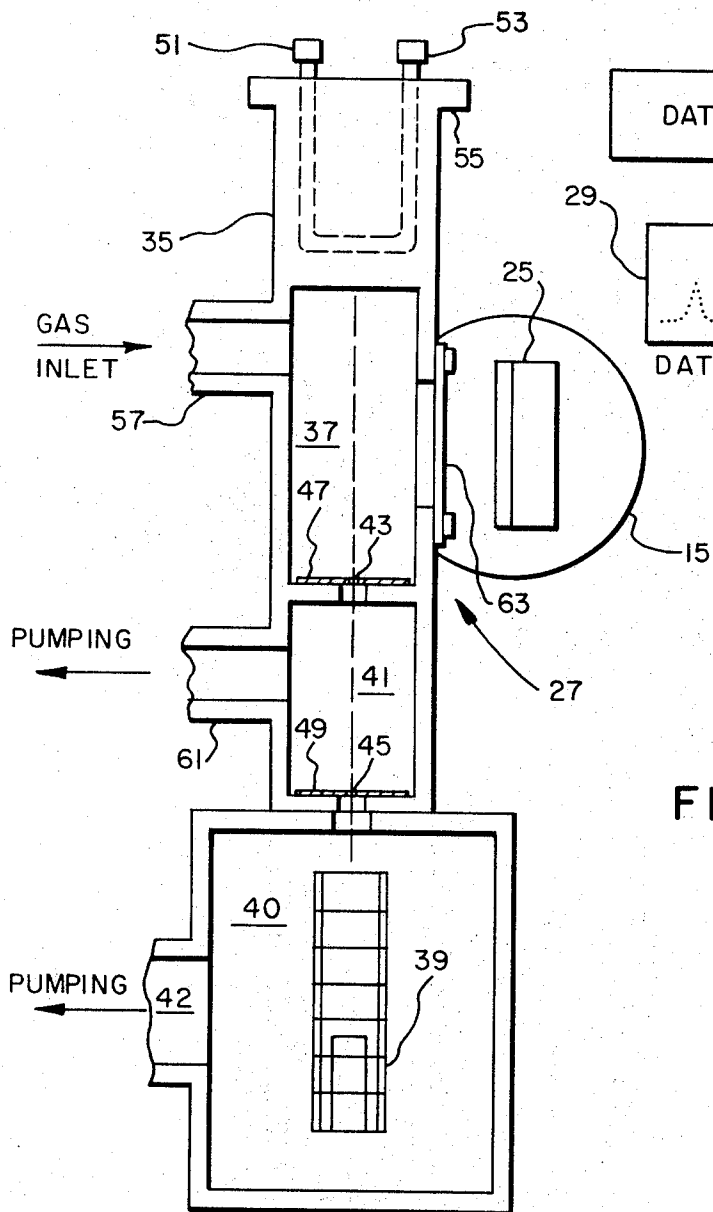


FIG. 2

ESCA X-RAY SOURCE

BACKGROUND OF THE INVENTION

This invention relates to electron spectroscopy for chemical analysis and more particularly to an essentially monochromatic X-ray source for irradiating samples to be analyzed.

The energy spectrum analysis of electrons obtained from an irradiated sample has proved a useful tool for chemical analysis. In general, useful electron spectra can be obtained by: (1) electron impact where the sample is irradiated with electrons so that the absorption spectrum is obtained or so that a spectrum of secondary electrons is generated; (2) ultraviolet irradiation where the irradiation produces the ejection of electrons from the valence shell of the sample; and (3) X-ray-induced photoelectron spectroscopy where the irradiation produces the ejection of electrons from inner shells of the sample atoms. The present invention relates to the latter form of electron spectroscopy.

As is understood by those skilled in the art, the resolution of the photoelectron energy spectrum obtained through X-ray irradiation is limited, not only by the inherent resolution of the electron analyzer, but also by the monochromaticity of the source providing the X-rays which irradiate the sample. Heretofore, most X-ray sources for electron spectroscopy have employed conventional metal targets followed by a filter for minimizing background radiation and selecting the characteristic X-ray line of principal interest.

In an attempt to obtain greater monochromaticity in the X-rays used for sample irradiation, constructions have been proposed in which an X-ray monochromator is interposed between the X-ray source and the sample. Such monochromators may, for example, employ crystal diffraction to spatially separate X-rays of different energies. However, the use of a monochromator necessarily entails a substantial loss in source intensity so that system sensitivity suffers correspondingly, even when substantially higher initial input powers are employed.

In accordance with one aspect of the present invention, it has been determined that substantially improved resolution can be obtained by employing a gas phase target in the X-ray source. In the X-ray source of the present invention, a gas is excited by an electron beam at energies sufficient to eject electrons from an inner shell of the target gas atom. A preferred target gas is neon at a pressure of 10^{-2} to 10^0 atmospheres with the electron beam possessing sufficient energy to excite the $K\alpha$ emission line.

In order to obtain electrons at sufficiently high energy levels to obtain the desired result, the present invention further contemplates an electron gun located in a chamber separate from the target gas chamber, with the electrons being tightly focused through a small aperture between the gun and target chambers and with sufficient differential pumping being provided to maintain a vacuum around the electron gun, i.e., in the order of 10^{-5} torr.

Among the several objects of the invention then may be noted the provision of electron spectroscopy apparatus of improved resolution; the provision of such apparatus employing an X-ray source of essentially monochromatic nature; the provision of such apparatus which is highly reliable and which is of relatively simple and inexpensive construction. Other objects and fea-

tures will be in part apparent and in part pointed out hereinafter.

SUMMARY OF THE INVENTION

Briefly, the X-ray source of the present invention is adapted to irradiate a sample in an electron spectrometer which analyzes the energy distribution of photoelectrons given off by the sample in response to such irradiation. The source involves a target chamber for containing a gas at a preselected substantial pressure and, in a chamber separate from the target chamber, an electron gun directing a narrow beam of electrons into the target chamber, the beam energy being above that required to eject electrons from an inner shell of the target gas atoms. The electron beam passes through a relatively small aperture between the chambers and the electron gun chamber is pumped to maintain a vacuum which will allow the electrons to be accelerated to the required energies. X-rays given off by the target gas pass through a window in the target chamber and irradiate a sample at the entrance of the electron spectrometer.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a schematic diagram of ESCA apparatus employing the X-ray source of the present invention; and

FIG. 2 is a sectional view substantially on the line 2-2 of FIG. 1, showing in greater detail the construction of the X-ray source of the present invention.

Corresponding reference characters indicate corresponding parts throughout the several views of the drawings.

DESCRIPTION OF THE PREFERRED EMBODIMENT

Referring now to FIG. 1, there is illustrated at 10 electron energy spectroscopic apparatus analyzer of essentially conventional arrangement. This overall instrument may, for example, be the ESCA 36 model photoelectron spectrometer manufactured by the McPherson Instrument Corporation of Acton, Massachusetts which is described by John F. Rendina in an article entitled *Electron Spectroscopy For Chemical Analysis* appearing in the February, 1972 issue of *American Laboratory*. A pair of concentric spherical electrodes 11 and 13 form a double-focusing electron energy analyzer. By applying potentials of opposite polarity to the electrodes 11 and 13, electrons entering the space between the spheres through an entrance slit 15 can be caused to be focused on an exit slit 17, provided the energies of the electrons correspond to the applied potentials. A detector 19 measures the electron flux at the exit slit. By energizing the electrodes through a suitable scanning power supply, as indicated at 21, an energy spectrum is obtained which reflects the distribution of electron energies. As is conventional, the output signal obtained from the detector 19 is displayed and recorded, as indicated generally at 29, as a function of electron energy, the scanning and display being correlated by a suitable control unit as indicated generally at 31.

For chemical analysis purposes, the electrons introduced into the analyzer at the entrance slit 15 are typically obtained from a sample, as indicated at 25, which is irradiated to generate the electron spectrum. In accordance with the present invention, the photoelec-

trons are obtained by irradiating the sample 25 with X-rays provided by a source constructed in accordance with the present invention, the source being indicated generally at 27. As is conventional, the sample chamber is pumped to maintain a relatively high vacuum, i.e., in the order of 10^{-7} torr.

Referring now to FIG. 2, the X-ray source 27 involves a relatively heavy metal housing 35, shaped to provide a generally cylindrical target chamber 37. Aligned with the cylindrical target chamber 37 is a high energy electron gun 39, located in a chamber of its own, designated 40. The gun chamber 40 is provided with a port 42 through which the chamber is pumped to maintain the necessary vacuum around the gun. The electron gun 39 may, for example, be of the type typically employed for electron welding purposes, i.e., one of which will generate a relatively tightly focused electron beam of up to 50 milliamperes with energies up to 20 kilovolts. A beam spot size of 0.020 inches may be considered typical.

The electron beam generated by the gun 39 is directed into the target chamber 37 through a differential pumping chamber 41, the beam passing through small apertures 43 and 45 in the walls 47 and 49 between the various chambers. Preferably, both the walls are constructed as thin diaphragms, as illustrated. With such a construction, the apertures 43 and 45 can be cut by the electron gun itself, thereby obtaining inherent alignment. The upper end wall of the target chamber preferably constitutes an electron trap and heat sink, as indicated, with suitable water cooling ports 51 and 53 and a flange 55 by means of which the target assembly may be mounted.

An inlet 57 is provided to the target chamber 37. Through this port, a suitable target gas is provided at a pressure of about 10^{-2} to 10^0 atmosphere, i.e., a very substantial pressure as compared with the vacuum normally provided in almost all parts of an electron spectrometer. For reasons explained hereinafter, a preferred target gas is neon, the excitation of the neon by the electron beam being at an energy sufficient to excite the $K\alpha$ transition of the neon atom. As is understood, this transition involves removal of electrons from the inner shell of the gas atom.

In that the pressure within the target chamber 37 is substantially higher than that maintained in the gun chamber 40, some of the target gas will continuously leak out of the target chamber through the aperture 43. While the environment of the electron gun 39 is continuously pumped as described previously, it is preferable to also apply independent pumping to the differential pumping chamber, i.e., through a suitable port 61, so as to minimize the escape of the target gas into the sample chamber and to minimize collisions between the electron beam and the target gas other than those occurring within the target chamber itself. Pumping to a level of about 10^{-1} torr is suitable. As is understood, the electron gun 39 must operate in a substantially evacuated environment, e.g. 10^{-5} torr, in order to accelerate electrons up to the desired energy levels, i.e., up to 20 kilovolts. The presence of any substantial gas pressure around the gun would render it impossible to achieve such energies since intervening collisions would absorb energy and produce arcing, preventing the gun from operating properly and thus the gun chamber is pumped separately from the differential pumping chamber. In some cases, the electron gun may

be located in the sample chamber where a high vacuum is maintained.

From the foregoing, it can be seen that this construction permits a high energy electron beam to be impinged upon a suitable target gas, while the gas is maintained at relatively high pressure. As the electrons constituting the beam generated by gun 39 possess sufficient energy to excite the $K\alpha$ transition of the preferred target gas, neon, X-rays will be given off by the impingement of the electron beam on the target gas. Further, since the impingement occurs mainly in the target chamber 37, where the gas is at substantial pressure as compared with the sample chamber, most of the X-rays will be generated within the chamber 37. A thin window 63 of a material such as aluminum or beryllium permits these X-rays to escape the chamber 37 and irradiate the sample 25 located adjacent the target chamber. As is understood by those skilled in the art, the window 63 is selected to absorb any secondary lines and background radiation which may be present, though with the gaseous neon target these components are reduced as compared with a conventional metallic target. As described previously, the sample will give off photoelectrons in response to the X-ray irradiation, the energy spectrum of this photoelectron emission being analyzed as illustrated in FIG. 1.

As compared with the usual metallic X-ray target, the gas target of the present invention provides X-rays of relatively high monochromaticity. The improved monochromaticity of the X-ray energy used for irradiating the sample 25 permits much greater resolution in the photoelectron energy spectrum analysis subsequently performed and thus greater accuracy can be obtained in chemical analysis using the overall system of the present invention.

While the neon $K\alpha$ emission line is the presently preferred source, it should be understood that other gases and other transitions could be utilized in the practice of this invention.

In view of the foregoing, it may be seen that several objects of the present invention are achieved and other advantageous results have been attained.

As various changes could be made in the above constructions without departing from the scope of the invention, it should be understood that all matter contained in the above description or shown in the accompanying drawings shall be interpreted as illustrative and not in a limiting sense.

What is claimed is:

1. An electron spectrometer comprising:
 - means for holding a sample which is to be analyzed;
 - means for analyzing photoelectrons given off by said sample as a function of their energies thereby to obtain an energy spectrum;
 - housing means defining an X-ray target chamber;
 - means for providing neon gas to said chamber at substantial pressure, said housing means also defining a chamber separate from said target chamber, there being a small aperture between said chambers;
 - in said separate chamber, an electron gun for directing, through said small aperture a narrow beam of electrons into said target chamber at an energy level above that required to excite the neon $K\alpha$ emission line;
 - means for pumping the electron gun chamber to maintain a vacuum in the presence of gas flow

5

6

through said aperture; and an X-ray window in said target chamber allowing X-rays comprising mainly the $K\alpha$ neon emission line to essentially directly irradiate said sample without intervening dispersive selection thereby to obtain from said analyzing means a spectrum of improved resolution.

2. Apparatus as set forth in claim 1 wherein said neon gas is provided at a pressure of about 10^{-2} - 10^0 atmospheres.

3. Apparatus as set forth in claim 1 wherein one end of said target chamber comprises a water-cooled elec-

tron trap.

4. Apparatus as set forth in claim 1 wherein each end of said differential pumping chamber comprises a diaphragm-like member having one of said small apertures formed by said electron gun.

5. Apparatus as set forth in claim 1 wherein said window is aluminum.

6. Apparatus as set forth in claim 1 wherein said window is beryllium.

* * * * *

15

20

25

30

35

40

45

50

55

60

65