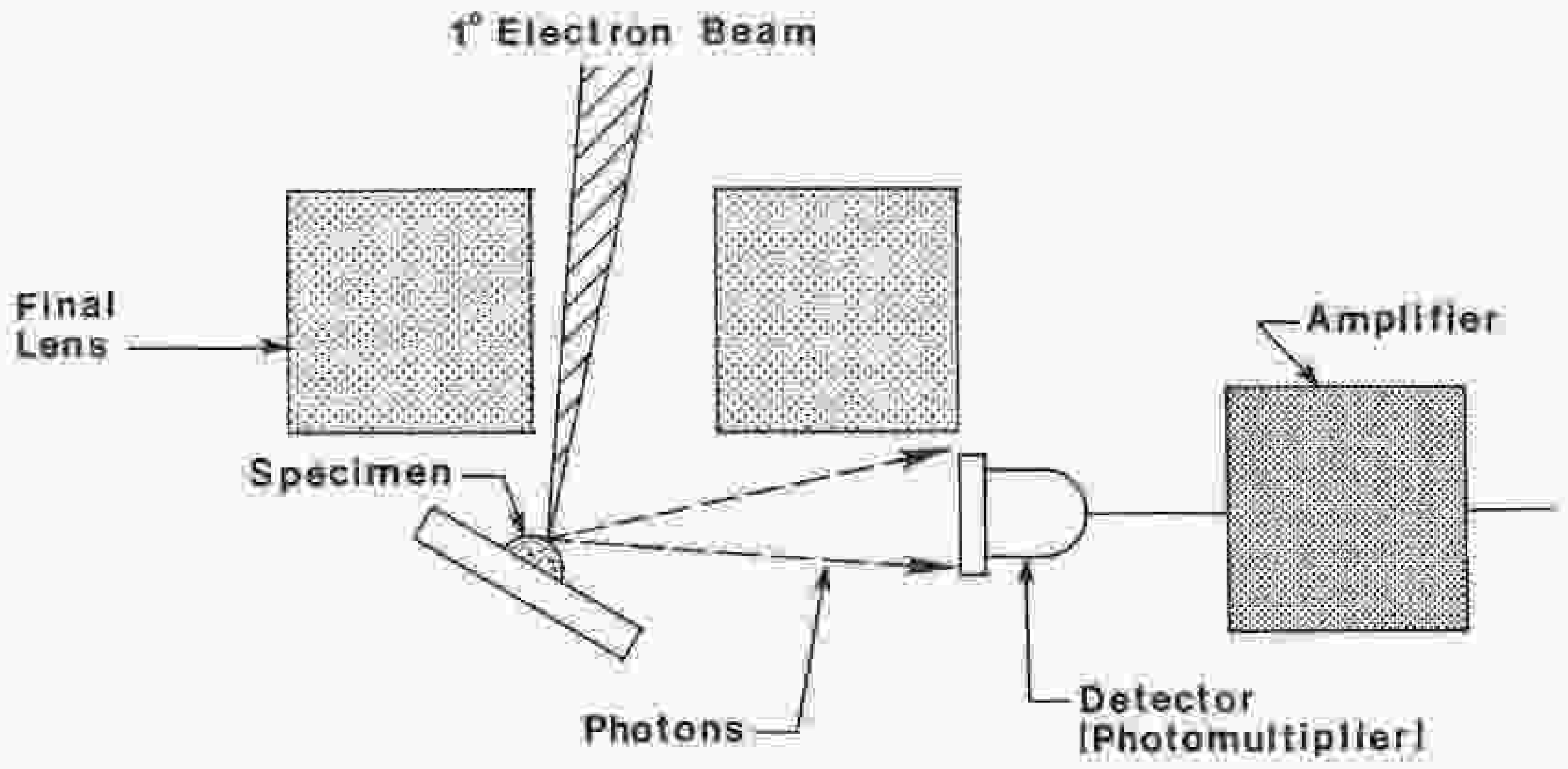


3-16. Block diagram of one type of cathodoluminescence detector in a scanning electron microscope.

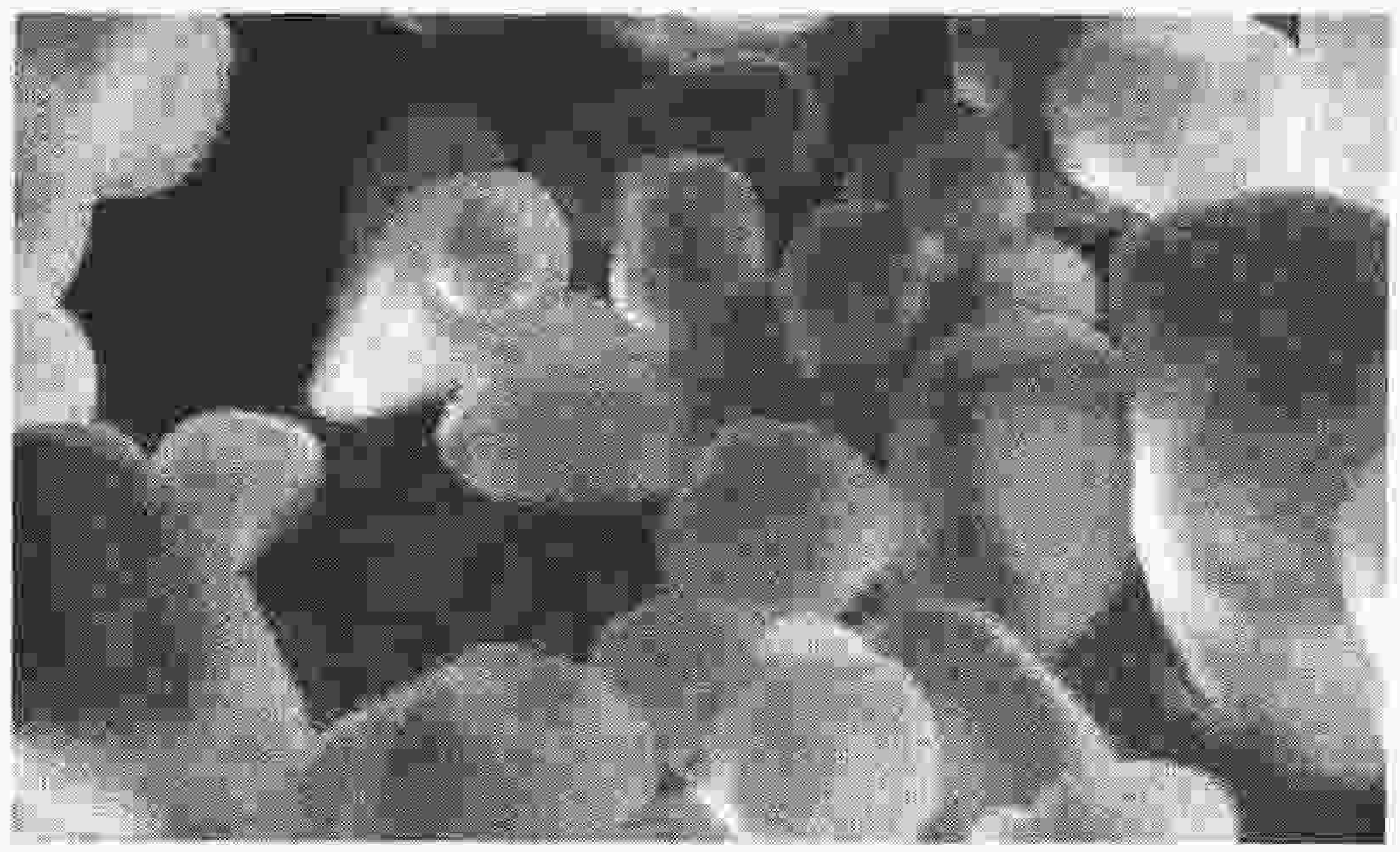
photomultiplier tube for processing the emitted photons. This arrangement provides for interchangeable viewing of secondary electron and cathodoluminescence images. If a second cathode ray tube is present, the two images may be observed simultaneously. It is also possible to project the light emitted through a monochromator slit to measure the luminescent spectrum from a chosen portion of a cathodoluminescence image (24,39). Other detector designs, such as a collector turret have also been described (38).

The intensity of cathodoluminescence ranges from very weak outputs in plastics and glass up to about 10% of the absorbed energy in certain phosphors (15). Cathodoluminescence intensity is reduced by 50% at all wavelengths if a 20 nm carbon coating is used for conductivity. Methods to increase detection of this relatively weak signal have included improvement of the photomultiplier tube voltage supply circuit and installation of a half-parabolic mirror to increase collection efficiency (3). Spatial resolution has been estimated at 50 nm under qualified instrumental and specimen conditions (39 and Fig. 3-17).

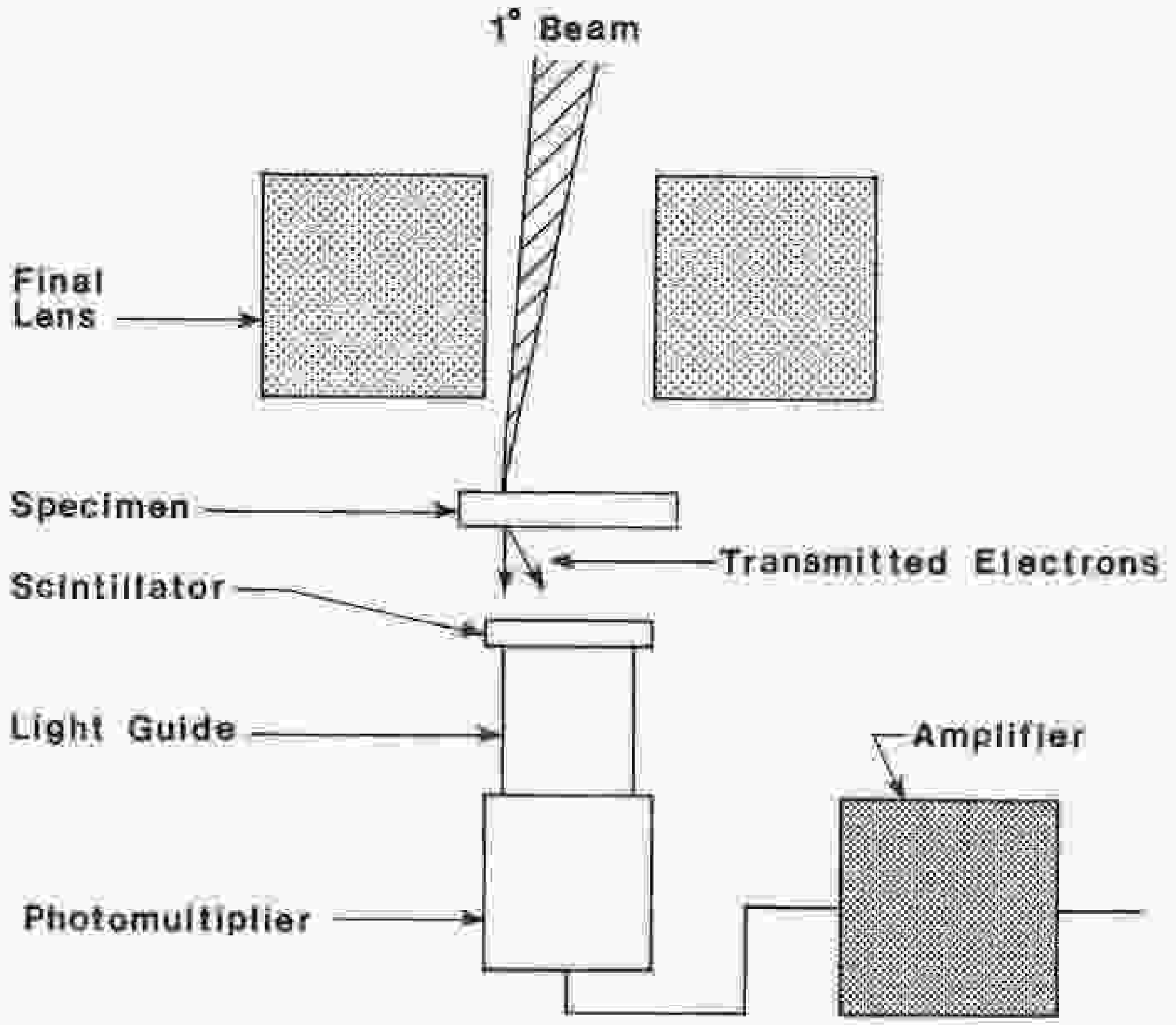
TRANSMITTED ELECTRONS

The placement of a detector system below a thin specimen ($< 1\ \mu$m) in a conventional SEM will result in the collection of those electrons having enough energy to escape from the underside of that specimen (Fig. 3-18). Since a detector so located will collect electrons over a wide range of energies, from that of beam electrons elastically scattered to those secondary electrons excited at the lower surface, the signal will be somewhat noisy and lower in resolution than its transmission electron microscope counterpart. For very thin specimens, 5 nm resolution or better is obtainable by scanning electron microscopes operated in this mode. For specimens nearing $1.0\ \mu$m, the resolution is decreased because the beam broadens as it passes through the specimen (29).

The transmitted electron detection mode offers information unique in SEM work by allowing the examination of internal specimen ultrastructure (Fig. 3-19). This examination may also be coupled with pre-existing x-ray microanalysis equipment for the acquisition of elemental information and distribution. Provided the equipment and time are available for preparing thin specimens, transmitted electron detection capabilities may be a valuable research asset.



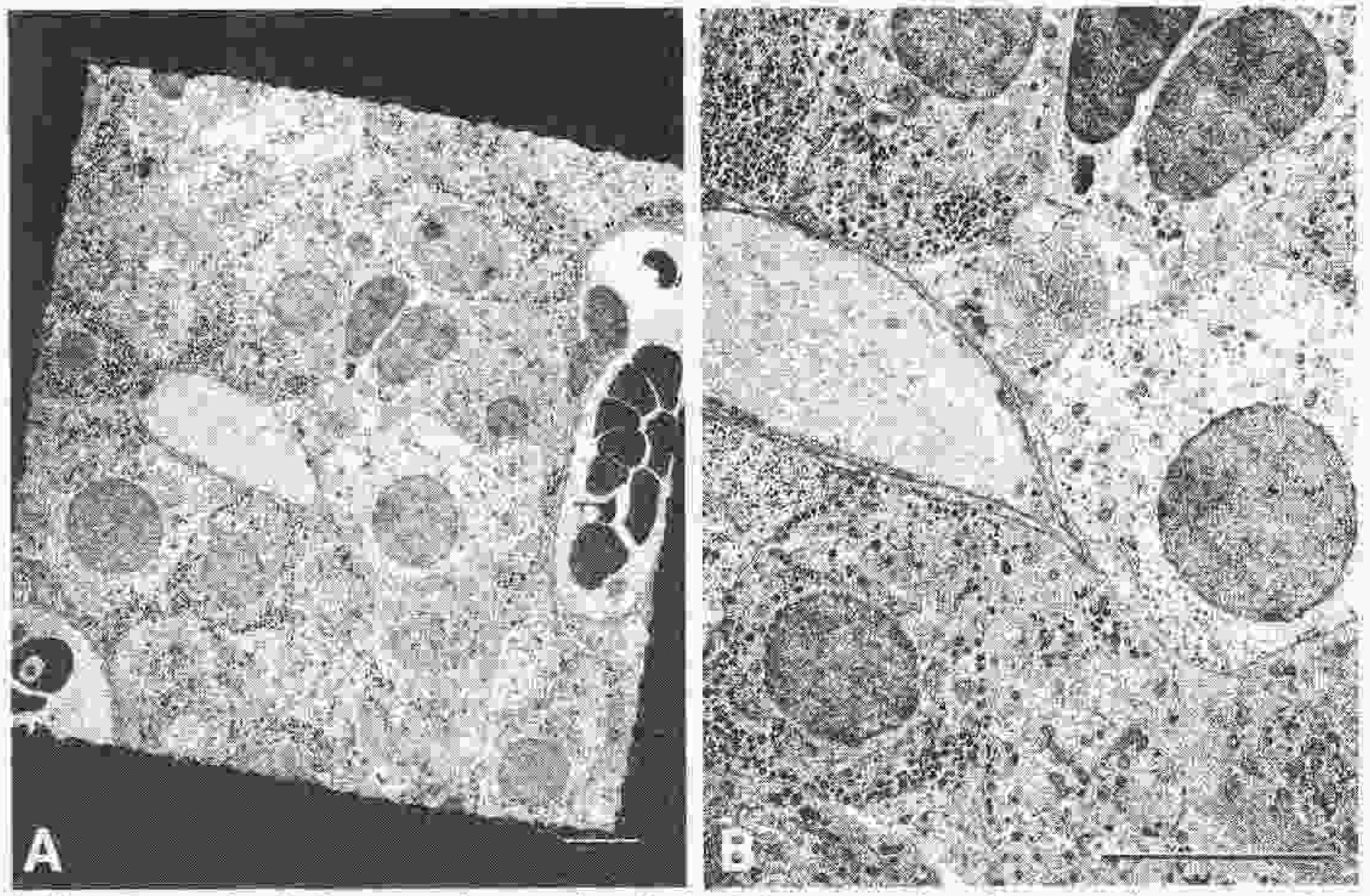
3-17. Cathodoluminescence image of a P-7 phosphor at 20 KeV. Line scale is equal to 50 μm (Courtesy of ETEC Corp.).



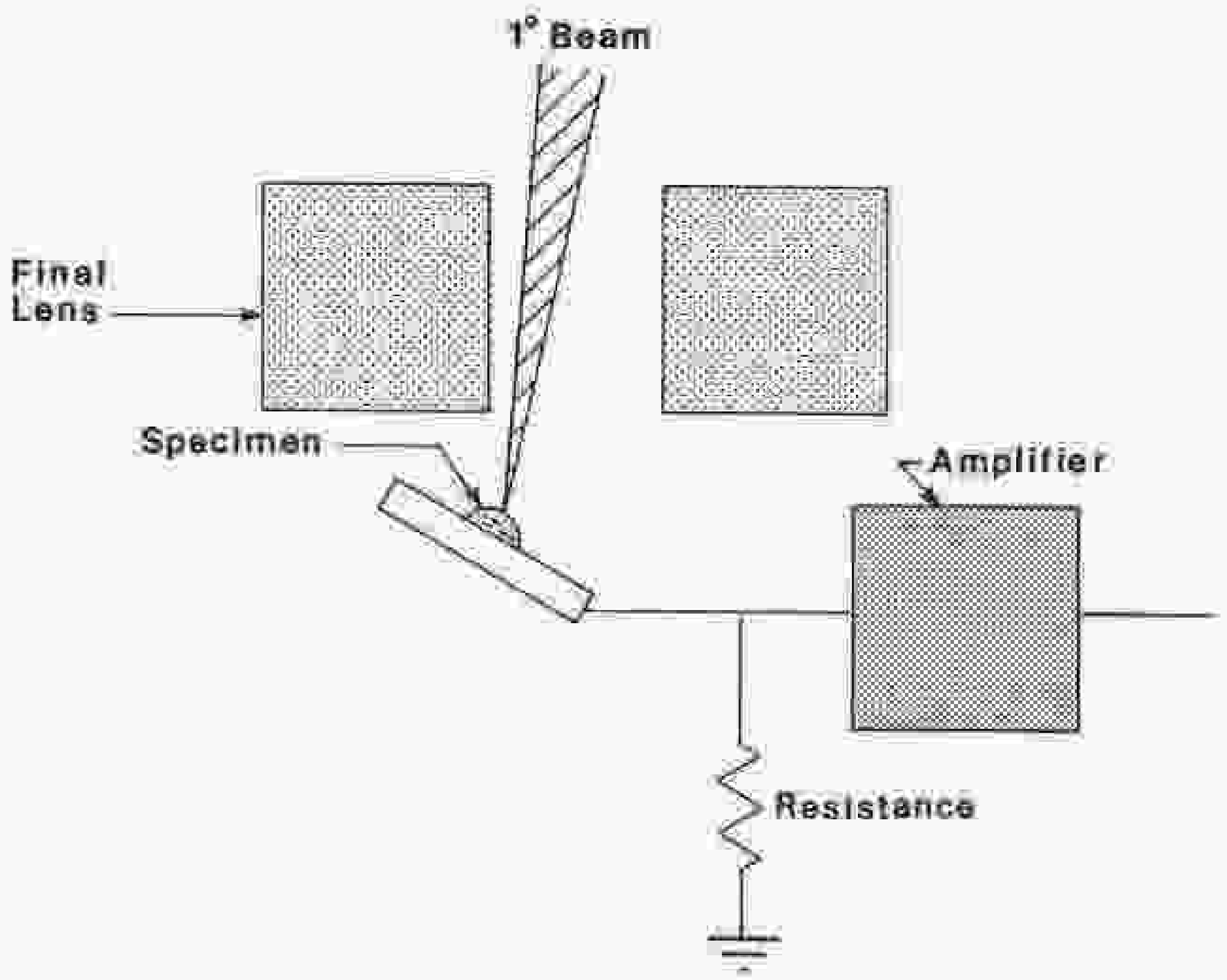
3-18. Block diagram of a transmitted electron detector system in a scanning electron microscope.

SPECIMEN CURRENT

The magnitude of specimen current produced for a given specimen is the difference between beam current and emissive current. The latter term is the sum of all currents leaving the specimen by emission of electrons. The change in current from point to point in the specimen may be imaged (25 and Fig. 3-20). It follows that a change in the intensity of backscattered or secondary electron signals would inversely affect the specimen current signal. Therefore, those specimens



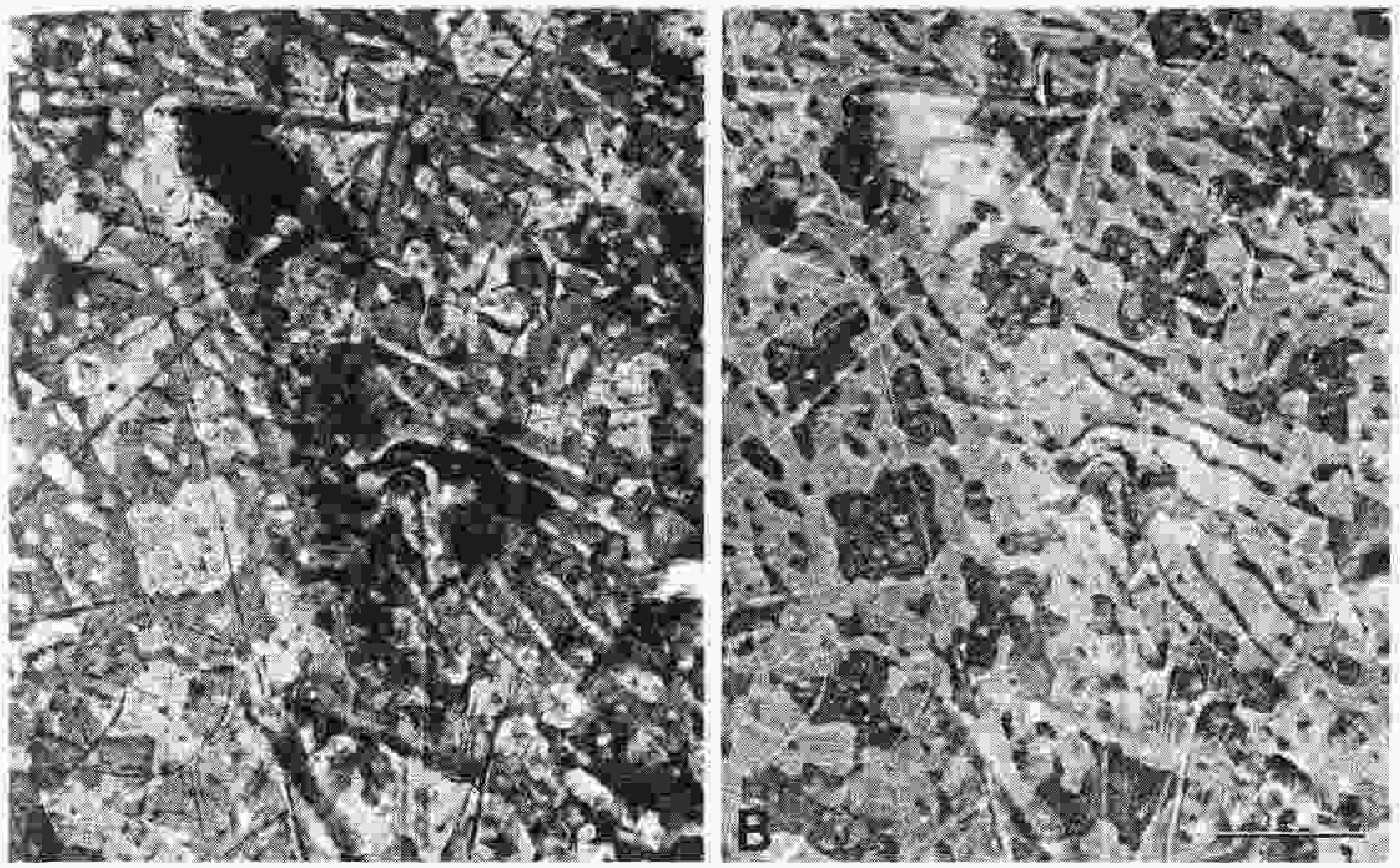
3-19. Transmitted electron micrographs obtained in a scanning electron microscope of rat pituitary gland. (A) Low magnification. (B) Higher magnification. Line scale equal to 4 μm (Courtesy of American Optical Corp.).



3-20. Block diagram showing one type of absorbed electron detector in a scanning electron microscope.

with stronger emissive signals would have weaker specimen current signals (Fig. 3-21).

This signal has the advantage of removing the detector-specimen orientation considerations because the signal is detected within the specimen itself. In studying large specimens, this signal is of particular value in being functional at very small working distances (8).



3-21. Specimen current detection of a wire surface. (A) Secondary electron image. (B) Specimen current image. Line scale is equal to 10 μm (Courtesy of Philips Corp.).

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CHAPTER FOUR

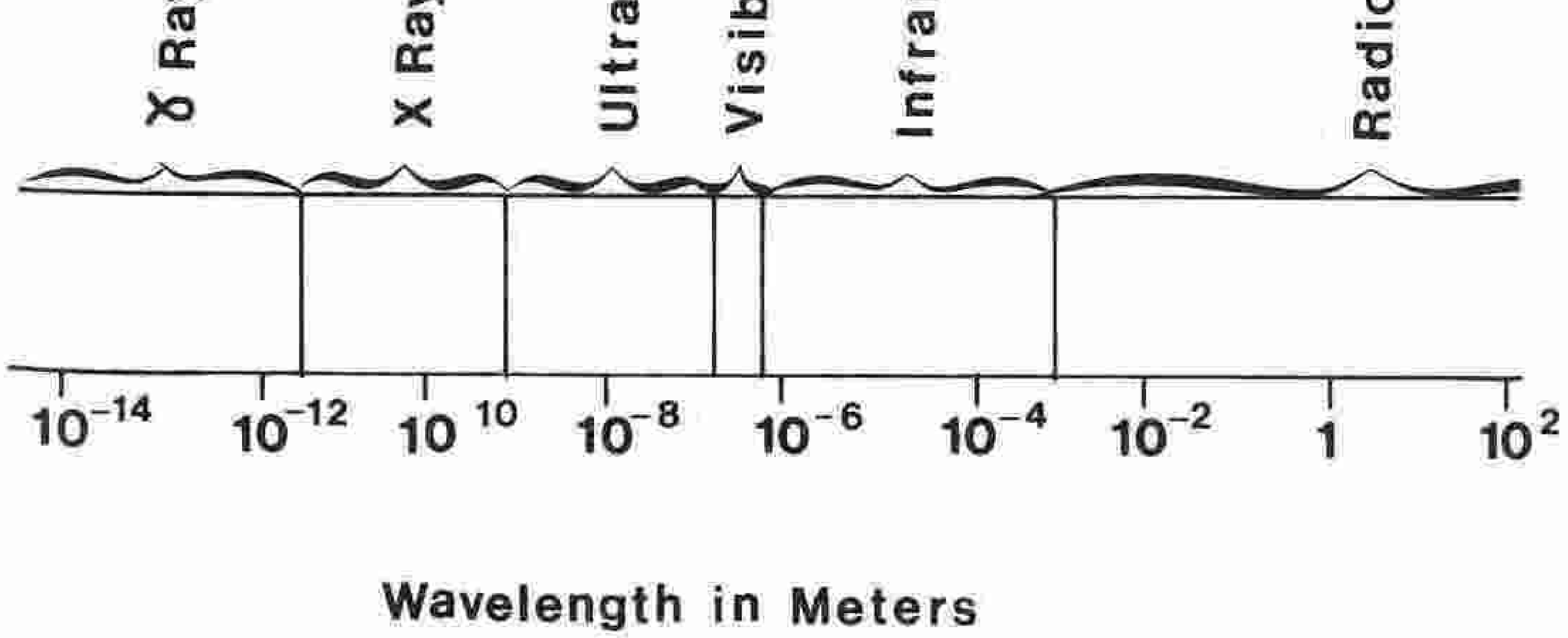
X-RAY ANALYSIS

The various signals produced when an electron beam impinges upon a sample in the scanning electron microscope have been discussed in previous chapters. High energy x-rays are one of the more widely used of these signals. X-ray analysis is widely referred to as a form of analytical electron microscopy and has been applied in a great variety of scientific and technical fields.

Analytical electron microscopy is accomplished by the coupling of an x-ray analysis device with (in this case) a scanning electron microscope. The unique combination that results can permit selected microscopic and macroscopic samples to be analyzed for elemental composition in the region traversed by the electron beam. This analysis can be obtained simultaneously with the visual representation or photograph of the identical area. The entire analysis can be performed quickly, often with minimal specimen preparation. More importantly, it is non-destructive to the sample.

THE X-RAY SPECTRUM

X-rays, like visible light, are a form of electromagnetic radiation. As can be seen in Figure 4-1, however, x-rays characteristically have a much shorter



4-1. The electromagnetic spectrum.

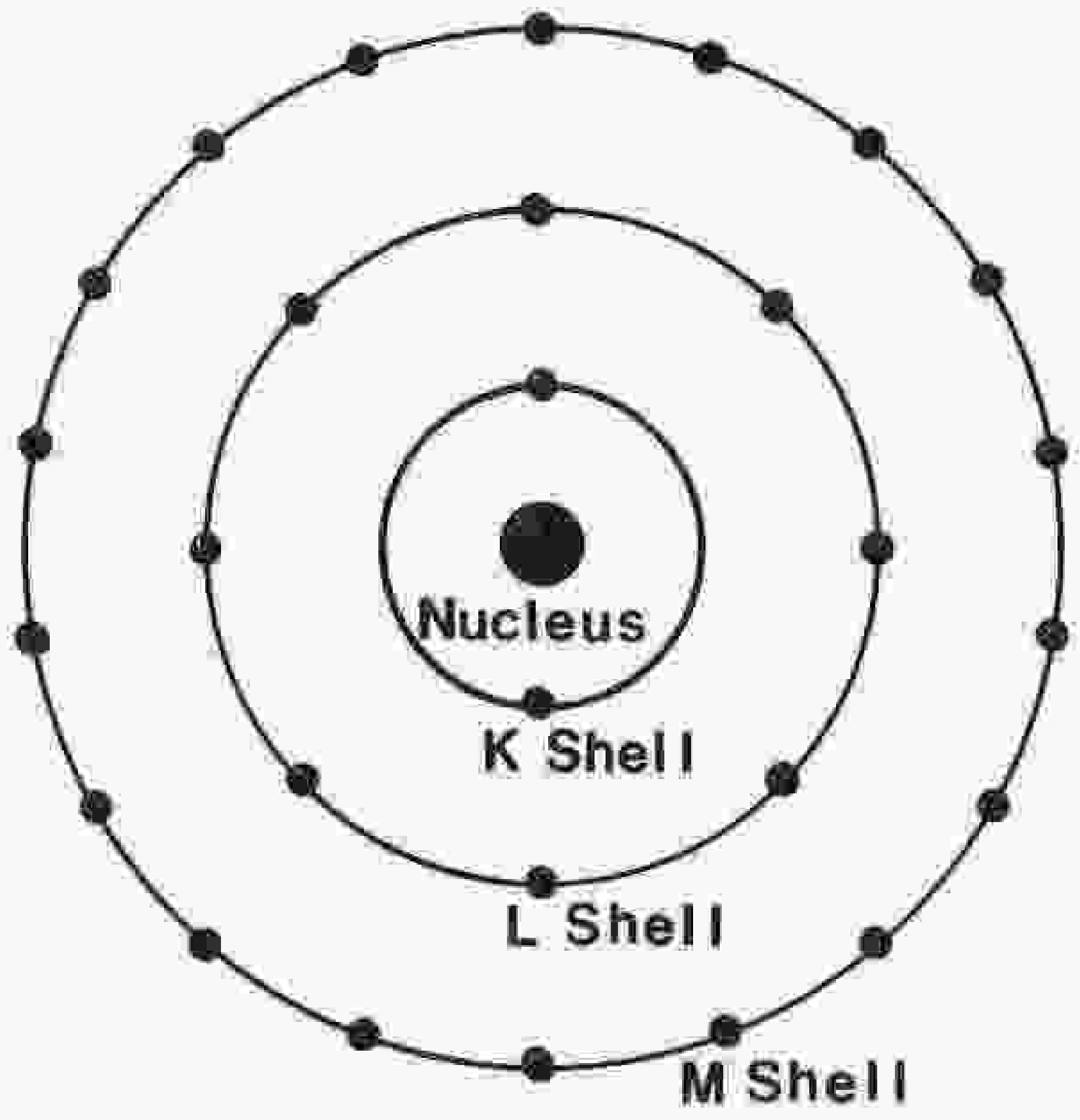
wavelength (0.01 to 10 nm) and a higher frequency. Initially, the term "x-ray" was applied to this type of radiation as its nature was unknown. It is now known that x-rays are created when an electron undergoes a decrease in energy.

Basic physics dictates that electrons of a given atom occupy distinct energy levels termed "shells" (Figure 4-2). By convention, the shell closest to the nucleus is termed the "K shell" and contains up to two electrons. The next shell from the nucleus is termed the "L shell" and contains up to eight electrons. Subsequent shells are the "M" with up to 18 electrons, "N" with up to 32 electrons, and so forth.

The number of electrons present in an atom is equal to the number of protons present in its nucleus (i.e., the atomic number commonly designated Z) and determines the number of shells present in a particular atom. The most stable atomic configuration requires that lower energy shells are filled first. Thus, a hydrogen atom (Z = 1) has a single electron which occupies a K shell. An atom of iron (Z = 26) has 2 electrons in the K shell, 8 electrons in the L shell, and 16 electrons in the M shell.

Electrons always seek the lowest possible energy level. Thus, the removal of an electron from a low energy inner shell will result in the immediate replacement by an electron from a higher energy outer shell. The second electron loses energy in this transfer (commonly referred to as an electron jump) and this energy is released as an x-ray whose energy is equal to the difference in energy between the two shells (Fig. 4-3).

A vacancy in an electron shell may be filled by an electron from any shell of greater energy, thus a spectrum of possible x-rays exists for each element. An alphanumeric system of nomenclature has been developed to identify these resultant x-rays. Each x-ray is first named for the shell which was initially vacated to create the x-ray. Thus, an x-ray which is created by the filling of a vacancy in a K shell is termed a K x-ray; the filling of an L shell creates an L x-ray. The x-ray is further distinguished by the size of the electron jump that created it. A vacancy filled by an electron from an adjacent shell creates an x-ray termed α. A difference of two shells creates a β x-ray. A difference of three shells creates a γ x-ray. Thus an electron that jumps from an L shell to a K shell creates a K_α x-ray; an electron jump from an N shell to an L shell creates an L_β x-ray (Fig. 4-4).

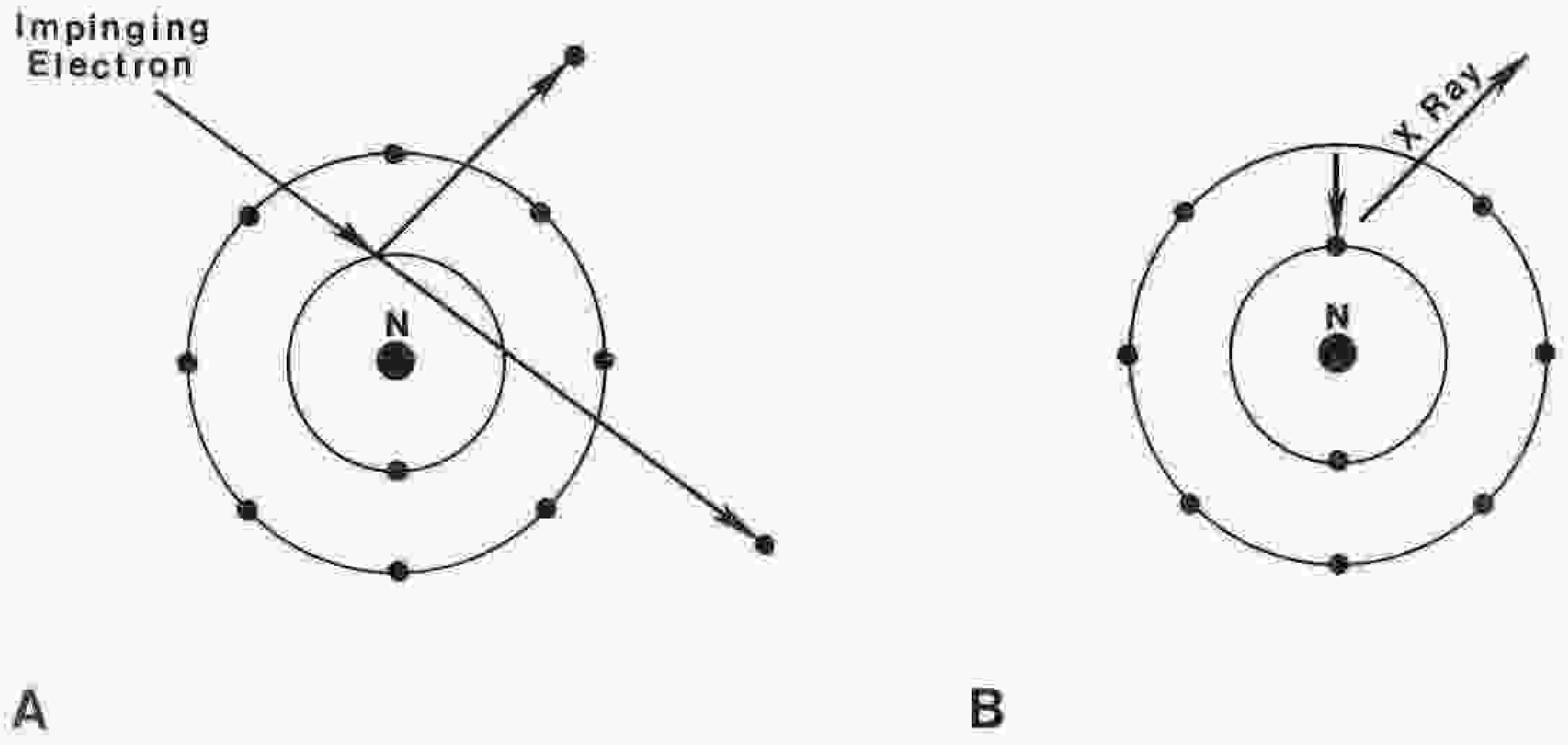


4.2 Schematic diagram of an atom showing the electron shells.

Differences in electron spin create slight differences in energy between electrons in the same shell and thus in the energy of x-rays emitted by electron jumps. There are characteristically two α energies termed α_1 and α_2; up to four β energies termed β_1, β_2, β_3 and β_4; and numerous γ energies. For many elements, the energy differences due to spin amount to less than 0.01% of the energy of either x-ray. For these x-rays the numerical subscript is usually eliminated entirely. For differences of up to several percent an average is often used and is so noted (i.e., $K_{\alpha_{av}}$).

The element iron (Z = 26) provides an example of a spectrum of x-ray energies. X-rays produced by electron jumps in iron atoms are shown below:

X-ray	Energy (KeV)
$Fe_{K_{\alpha_1}}$	6.403
$Fe_{K_{\alpha_2}}$	6.390
Fe_{K_β}	7.057
Fe_{L_α}	0.705
$Fe_{L_{\beta_{1\,\&\,2}}}$	0.718
$Fe_{L_{\beta_{3\,\&\,4}}}$	0.792
Fe_{L_γ}	0.721



4-3. Diagrammatic demonstration of the formation of an x-ray. (A) An impinging electron removing a specimen electron from a K-shell and (B) the vacancy being filled by an L-shell electron with the generation of an x-ray. The resultant x-ray would be a K_α x-ray.