

Fig. 7-8 Arrangement of slits in diffractometer.

Because of the focusing of the diffracted rays and the relatively large radius of the diffractometer circle, about 15 cm in commercial instruments, a diffractometer can resolve very closely spaced diffraction lines. Indicative of this is the fact that resolution of the Cu $K\alpha$ doublet can be obtained at 2θ angles as low as about 40° . Such resolution can only be achieved with a correctly adjusted instrument, and it is necessary to so align the component parts that the following conditions are satisfied for all diffraction angles:

1. line source, specimen surface, and receiving-slit axis are all parallel,
2. the specimen surface coincides with the diffractometer axis, and
3. the line source and receiving slit both lie on the diffractometer circle.

7-4 COUNTERS (GENERAL)

Without exception all electronic counters were developed by nuclear physicists for studies of radioactivity. They can detect not only x - and γ -radiation, but also charged particles such as electrons and α -particles, and the design of the counter and associated circuits depends to some extent on what is to be detected. Here we are concerned only with counters for the detection of x -rays of the wavelengths commonly encountered in diffraction.

Four types of counters are currently in use: proportional, Geiger, scintillation, and semiconductor. All depend on the power of x -rays to ionize atoms, whether they are atoms of a gas (proportional and Geiger counters) or atoms of a solid (scintillation and semiconductor counters). A general treatment of the first three types has been given by Parrish [7.8].

We will be interested in three aspects of counter behavior: losses, efficiency, and energy resolution. These are defined below and made more specific in later sections on particular counters.

Counting Losses

The absorption of a quantum (photon) of x-rays in the active volume of a counter causes a voltage pulse in the counter output. Pulses from the counter then enter some very complex electronic circuitry, consisting of one or more pulse amplifiers, pulse shapers, etc. and, at the end, a scaler or ratemeter and, possibly, a pulse-height analyzer (Sec. 7-9). Let us call all the circuitry beyond the counter simply the "electronics." Then we are interested not simply in the behavior of the counter alone, but in the behavior of the whole system, namely, the counter-electronics combination.

If the x-ray beam to be measured is strong, the rate of pulse production in the counter will be high, and the counting rate given by the ratemeter will be high. (Roughly speaking, several thousand counts per second is a "high" rate in powder diffractometry, and less than a hundred cps a "low" rate.) As the counting rate increases, the time interval between pulses decreases and may become so small that adjacent pulses merge with one another and are no longer resolved, or counted, as separate pulses. At this point counting loss has begun. The quantity that determines this point is the *resolving time* t_s of the counter-electronics system, defined as the minimum time between two resolvable pulses.

The arrival of x-ray quanta at the counter is random in time. Therefore pulse production in the counter is random in time, and a curve showing the change in voltage of the counter output would look like Fig. 7-9. If the arrival and absorption of entering quanta were absolutely periodic in time, the maximum counting rate without losses would be given simply by $1/t_s$. But even if their average rate of arrival is no greater than $1/t_s$, some successive quanta may be spaced less than t_s apart because of their randomness in time. It follows that counting losses will occur at rates less than $1/t_s$ and that losses will increase as the rate increases, as shown in Fig. 7-10. Here "quanta absorbed per second" are directly proportional to the x-ray intensity, so that this curve has an important bearing on diffractometer measurements, because it shows the point at which the observed counting rate is no longer proportional to the x-ray intensity. The straight line shows the ideal response that can be obtained with a proportional counter at the rates shown. This linear, no-loss behavior is fortunately typical of most counters used today in diffractometry; otherwise one would have the tedious task of correcting some observed counting rates for losses.



Fig. 7-9 Randomly spaced voltage pulses produced by a counter.

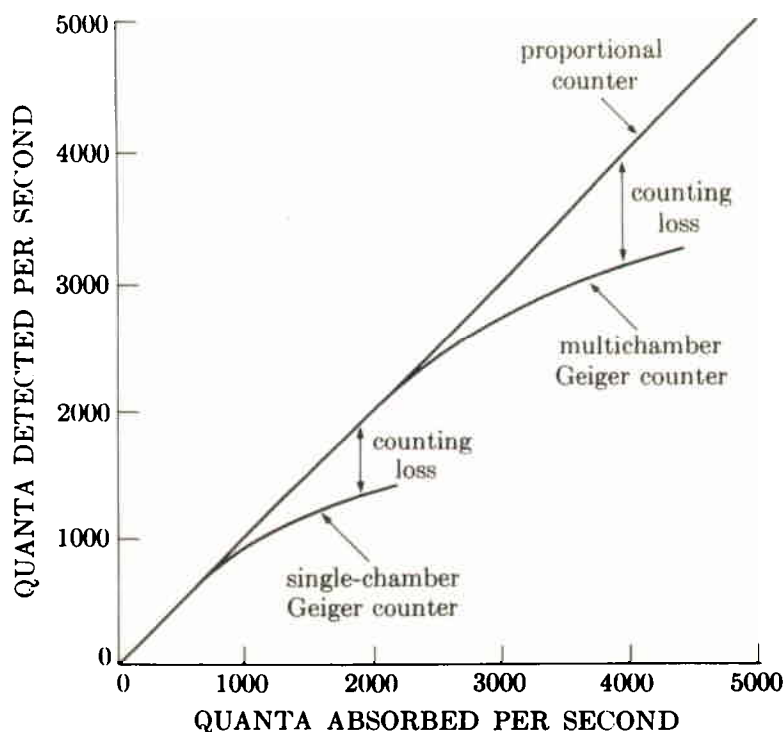


Fig. 7-10 The effect of counting rate on counting losses for three kinds of counter (schematic).

If the resolving time t_s of the counter-electronics is known, the point at which losses begin can be calculated by an easily remembered rule: a loss of one percent occurs at a rate of about one percent of $1/t_s$. Thus, if t_s is one microsecond, the counting rate should be linear to within one percent up to a rate of about 10,000 cps.

Ordinarily, the resolving time is unknown. But if nonlinear counting behavior is suspected, the counting rate at which losses begin can be determined experimentally by the following procedure. Position the counter to receive a strong diffracted beam, and insert in this beam a sufficient number of metal foils of uniform thickness to reduce the counting rate almost to the cosmic background. (Cosmic rays, because of their high penetrating power, pass right through the walls of the counter and continually produce a few counts per second.) Measure the counting rate, remove one foil, measure the counting rate, and continue in this manner until all the foils have been removed. Because each foil produces the same fractional absorption of the energy incident on it, a plot of observed counting rate (on a logarithmic scale) vs. number of foils removed from the beam (on a linear scale) will be linear up to the point where losses begin and will in fact resemble Fig. 7-10. A curve of this kind is shown in Fig. 7-11.

Counting Efficiency

The overall efficiency E of the counter-electronics system in detecting incident x-ray quanta as resolved pulses is the product of the absorption efficiency E_{abs} and the detection efficiency E_{det} .

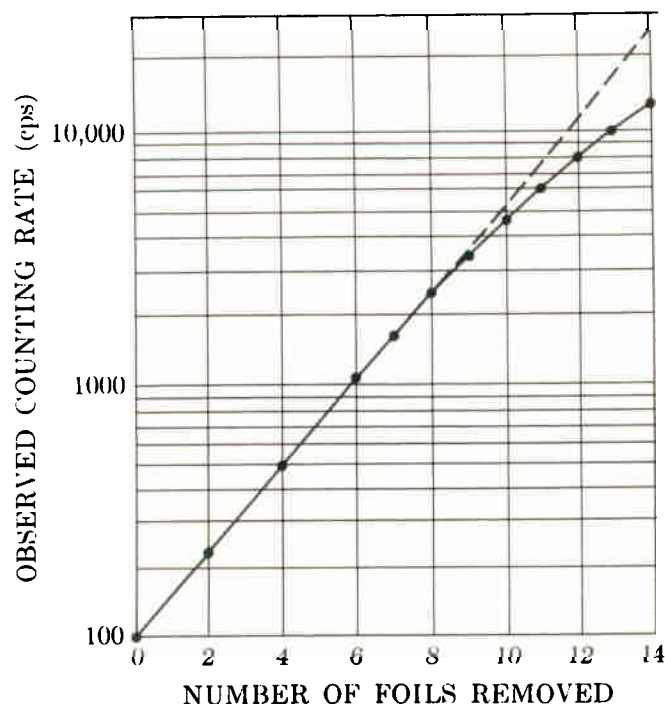


Fig. 7-11 Calibration curve of a multichamber Geiger counter. Cu $K\alpha$ radiation. Nickel foils, each 0.01 mm thick, used as absorbers.

All counters have a thin "window," usually of mica or beryllium, through which the x-rays must pass before reaching the active volume of the counter. The fraction of the incident radiation absorbed by the window $f_{\text{abs}, w}$ should be as small as possible, and the fraction absorbed by the counter itself $f_{\text{abs}, c}$ as large as possible. The absorption efficiency E_{abs} , expressed as a fraction, is given by $(1 - f_{\text{abs}, w})(f_{\text{abs}, c})$. The detection efficiency E_{det} is simply $(1 - f_{\text{losses}})$, where f_{losses} represents the fractional counting losses described above. The overall efficiency is then

$$E = E_{\text{abs}}E_{\text{det}} = [(1 - f_{\text{abs}, w})(f_{\text{abs}, c})][1 - f_{\text{losses}}]. \quad (7-1)$$

As previously mentioned E_{det} is essentially 100 percent for most counters used in diffractometry. Therefore E is determined by E_{abs} , which can be calculated from the dimensions and absorption coefficients of the window and counter, and Fig. 7-12 shows the result. Note particularly the dependence of E_{abs} on wavelength, due to the dependence of absorption coefficients on wavelength. The efficiency of any counter is low for very short wavelengths, because most of these hard x-rays pass right through window and counter and are absorbed by neither; at long wavelengths E_{abs} decreases because of increasing absorption of soft x-rays by the window.

Energy Resolution

In most counters the size of the voltage pulse produced by the counter is proportional to the energy of the x-ray quantum absorbed. Thus, if absorption of a Cu $K\alpha$ quantum ($\lambda = 1.54 \text{ \AA}$, $h\nu = 9 \text{ keV}$) produces a pulse of V volts, then absorption of a Mo $K\alpha$ quantum ($\lambda = 0.71 \text{ \AA}$, $h\nu = 20 \text{ keV}$) will produce a pulse of $(20/9)V = 2.2 V$.

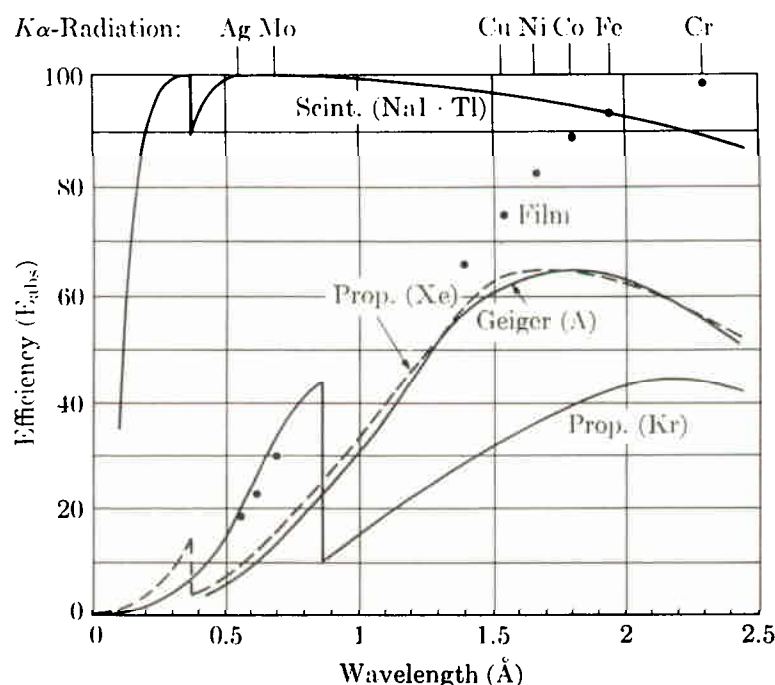


Fig. 7-12 Calculated values of absorption efficiency E_{abs} (in percent) of various kinds of counters and of photographic x-ray film (black dots). Parrish [7.8].

However, the size of a pulse is not sharply defined, even when the incident radiation is strictly monochromatic or, in energy terms, “monoenergetic.” Instead of all pulses having exactly the same size V as suggested, for example, by Fig. 7-9, they have sizes distributed around V roughly as indicated in Fig. 7-13. Here the ordinate, “counting rate,” is equivalent to the number of pulses having a particular size, so that this curve is a pulse-size distribution curve. If the width of the curve at half its maximum height is W and if V is the mean pulse size, then the resolution R of the counter is

$$R = \frac{W}{V} . \quad (7-2)$$

The smaller R , the better the resolution.

We will now examine the operation and performance of various counters.

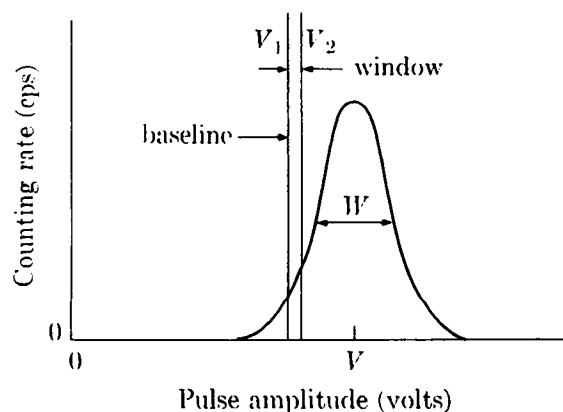


Fig. 7-13 Distribution curve of pulse size. (The “window” and “baseline” are explained in Sec. 7-9.)

7-5 PROPORTIONAL COUNTERS

Consider the device shown in Fig. 7-14, consisting of a cylindrical metal shell (the cathode), about 10 cm long and 2 cm in diameter, filled with a gas and containing a fine metal wire (the anode) running along its axis. Suppose there is a constant potential difference of about 200 volts between anode and cathode. One end of the cylinder is covered with a window of high transparency to x-rays. Of the x-rays which enter the cylinder, a small fraction passes right through, but the larger part is absorbed by the gas, and this absorption is accompanied by the ejection of photoelectrons and Compton recoil electrons from the atoms of the gas. The net result is ionization of the gas, producing electrons, which move under the influence of the electric field toward the wire anode, and positive gas ions, which move toward the cathode shell. At a potential difference of about 200 volts, all these electrons and ions will be collected on the electrodes, and, if the x-ray intensity is constant, there will be a small constant current of the order of 10^{-12} amp or less through the resistance R_1 . This current is a measure of the x-ray intensity. When operated in this manner, this device is called an *ionization chamber*. It was used in the original Bragg spectrometer but is now obsolete for the measurement of diffracted x-rays because of its low sensitivity. It is still used in some radiation survey meters.

The same instrument, however, can be made to act as a *proportional counter* if the voltage is raised to the neighborhood of 1000 volts. A new phenomenon now occurs, namely, multiple ionization or "gas amplification." The electric-field intensity is now so high that the electrons produced by the primary ionization are rapidly accelerated toward the wire anode and at an ever-increasing rate of acceleration, since the field intensity increases as the wire is approached. The electrons thus acquire enough energy to knock electrons out of other gas atoms, and these in turn cause further ionization and so on, until the number of atoms ionized by the absorption of a single x-ray quantum is some 10^3 to 10^5 times as large as the number ionized in an ionization chamber. As a result of this amplification a veritable avalanche of electrons hits the wire and causes an easily detectable pulse of current in the external circuit. This pulse leaks away through

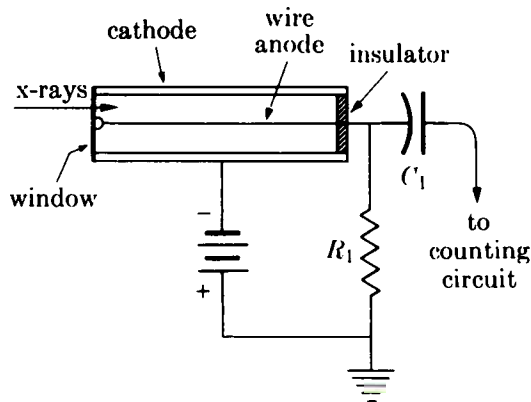


Fig. 7-14 Gas counter (proportional or Geiger) and basic circuit connections.

the large resistance R_1 but not before the charge momentarily added to the capacitor C_1 has been detected by the ratemeter or scaling circuit connected to C_1 . At the same time the positive gas ions move to the cathode but at a much lower rate because of their larger mass. This whole process, which is extremely fast, is triggered by the absorption of one x-ray quantum.

We can define a gas amplification factor A as follows: if n is the number of atoms ionized by one x-ray quantum, then An is the total number ionized by the cumulative process described above. (For example, if the gas in the counter is argon, energy of about 26 eV is required to produce an ion pair, i.e., a positive ion and an electron. If the incident radiation is Cu $K\alpha$ of energy 8040 eV, then the number n of ion pairs formed is 8040/26 or 310.) Figure 7-15 shows schematically how the gas amplification factor varies with the applied voltage. At the voltages used in ionization chambers, $A = 1$; i.e., there is no gas amplification, since the electrons produced by the primary ionization do not acquire enough energy to ionize other atoms. But when the voltage is raised into the proportional counter region, A becomes of the order of 10^3 to 10^5 , and a pulse of the order of a few millivolts is produced. Moreover, the size of this pulse is proportional to the energy of the x-ray quantum absorbed, which accounts for the name of this counter. This proportionality is important, because it allows us to distinguish (Sec. 7-9) between x-ray quanta of different energies (wavelengths). (Historically, this counter was the first kind to exhibit such proportionality. There are now others.) Pulses from the counter go to a preamplifier, mounted immediately adjacent to the counter; here they are amplified enough to be transmitted, without too much attenuation, along several feet of cable to the main amplifier and the rest of the electronics.

The correct voltage at which to operate the counter is found as follows. Position the counter to receive an x-ray beam of constant intensity. Measure the

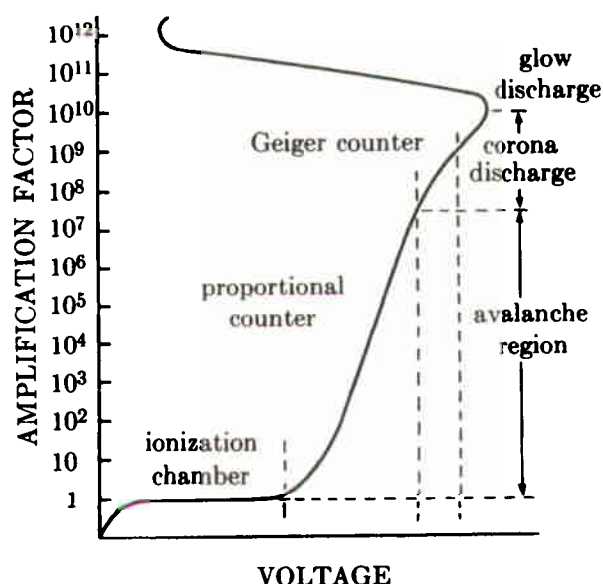


Fig. 7-15 Effect of voltage on the gas amplification factor. Friedman [7.9].

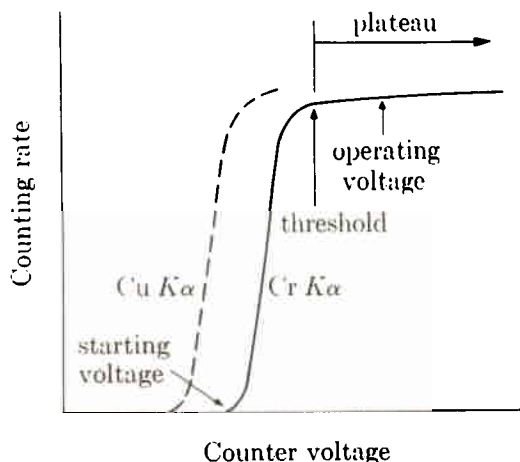


Fig. 7-16 Effect of voltage applied to proportional counter on observed counting rate at constant x-ray intensity (schematic).

counting rate with a ratemeter or scaler while slowly increasing the voltage applied to the counter from a low value. Figure 7-16 shows how the counting rate will vary with voltage. Below the starting voltage the pulse size is less than the input sensitivity of the counting circuit and no counts are observed. The pulse size and observed counting rate then increase rapidly with voltage up to the threshold of the plateau, where the counting rate is almost independent of voltage. The voltage is then fixed at about 100 volts above threshold. (Note that x-rays of longer wavelength require a higher counter voltage. This means that the counter voltage should be reset when the x-ray tube in the diffractometer is changed for one with a different target.)

The proportional counter is essentially a very fast counter and has a linear counting curve up to about 10,000 cps. This ability to separate closely spaced pulses is due to the fact that the avalanche triggered by the absorption of an x-ray quantum is confined to an extremely narrow region of the counter, 0.1 mm or less, and does not spread along the counter tube (Fig. 7-17). The rest of the counter volume is still sensitive to incoming x-rays.

The electric field near the end of the anode wire is not uniform. Most proportional counters are now made with a side window, rather than the end window shown in Fig. 7-17, so that x-ray absorption can take place in a region of uniform field.

The gas in the counter is usually xenon, argon, or krypton at a pressure somewhat less than atmospheric. Figure 7-12 shows that a krypton counter has about the same sensitivity for all the characteristic radiations normally used in diffraction. But an argon or xenon counter is much less sensitive to short wavelengths, an advantage in most cases. Thus, if a diffraction pattern is made with filtered radiation from a copper target, use of an argon counter will produce semi-monochromatic conditions, in that the counter will be highly sensitive to $\text{Cu } K\alpha$ radiation and relatively insensitive to the short wavelength radiation that forms the most intense part of the continuous spectrum. The diffraction background will therefore be lower than if a krypton counter had been used. (The

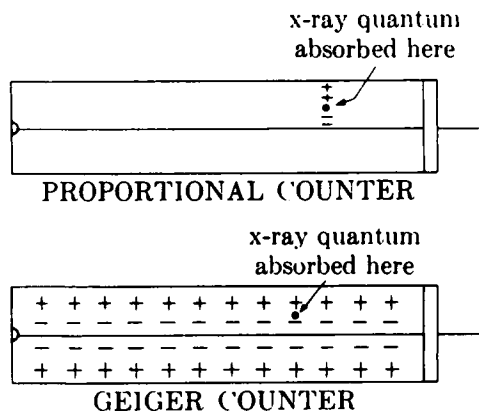


Fig. 7-17 Differences in the extent of ionization between proportional and Geiger counters. Each plus (or minus) symbol represents a large number of positive ions (or electrons).

student may wish to record the spectrum of the copper-target x-ray tube in a diffractometer. This can be done by operating it as a spectrometer, with a single crystal, such as quartz or rock salt, in the specimen holder and continuous scanning with a ratemeter and strip-chart recorder. With an argon-filled counter, the resulting spectrum will not look at all like what is expected (Fig. 1-5). Instead, only the $\text{Cu } K\alpha$ and $\text{Cu } K\beta$ lines will be visible on the chart, because the counter is insensitive to short wavelengths. The continuous spectrum can be observed only by effecting an opposite distortion: put several thicknesses of aluminum foil in the diffracted beam so as to absorb the $\text{Cu } K$ lines more than the short wavelengths; at the same time, expand the ratemeter scale to allow for the decreased intensity of all wavelengths. With sufficiently heavy filtration by aluminum, the spectrum can be so distorted that the maximum in the continuous spectrum will be more intense, as recorded, than the characteristic lines. It is an instructive experiment.)

In x-ray spectroscopy (Chap. 15), but not in diffraction, there is a need to measure soft x-rays of wavelength about $5\text{--}20 \text{ \AA}$. Because ordinary windows would almost totally absorb such radiation, thin sheet plastic is used as a window, so thin that it leaks. To allow for this, a stream of counter gas is continuously passed through the counter, which is then called a *gas-flow proportional counter* [G.29, 7-10].

Another special type, the *position-sensitive proportional counter* [7.11, 12, 13] may become important for certain applications. The diffracted x-ray beam enters the counter through a side window, striking the anode wire, which lies in the plane of the diffractometer circle, approximately at right angles. Because the electron avalanche is sharply localized (Fig. 7-17), the point where the electrons hit the wire can be determined by electronically measuring the time required for the pulse to travel from the point of impact to the end of the wire. Thus the angular position 2θ of a diffracted beam is found, not in the usual way by moving a counter with a narrow entrance slit to the position of the beam (Fig. 7-1), but by finding where the beam strikes the wire of a *fixed*, wide-window counter. The counting circuit must include a multichannel analyzer (Sec. 7-9) in order to determine the profile of the diffraction line. This new method is applicable only over a restricted range of 2θ values, but such a range is all that need be examined in some problems.

7-6 GEIGER COUNTERS

If the voltage on a proportional counter is increased to the neighborhood of 1500 volts, it will act as a Geiger counter. Historically, this was the first electronic counter; it is also called a Geiger-Müller or G-M counter.

The applied voltage is now so high that not only are some atoms ionized but others are raised to excited states and caused to emit ultraviolet radiation. These ultraviolet photons travel throughout the counter at high speed (light travels 10 cm in a third of a nanosecond), knocking electrons out of other gas atoms and out of the cathode shell. All the electrons so produced trigger other avalanches, and the net result is that one tremendous avalanche of electrons hits the whole length of the anode wire whenever an x-ray quantum is absorbed anywhere in the tube (Fig. 7-17). As a result the gas amplification factor A is now much larger, about 10^8 to 10^9 , than in a proportional counter, and so is the size of the pulse produced, now some 1 to 10 volts. This means that no preamplifier is needed at the counter. On the debit side, all pulses have the same size, whatever the energy of the x-ray quanta.

The Geiger counter is also slow. Any one avalanche of electrons hits the anode wire in less than a microsecond, but the slowly moving positive ions require about 200 microseconds to reach the cathode. Thus the electron avalanche leaves behind it a cylindrical sheath of positive ions around the anode wire. The presence of this ion sheath reduces the electric field between it and the wire below the threshold value necessary to produce a Geiger pulse. Until this ion sheath has moved far enough away from the wire, the counter is insensitive to entering x-ray quanta. If these quanta are arriving at a very rapid rate, it follows that not every one will cause a separate pulse and the counter will become "choked." The resolving time is only about 10^{-4} sec, so that counting losses begin at a few hundred cps. Even the multichamber counter is not much better (Fig. 7-10); this counter has a number of chambers side by side, each with its own anode wire, and one chamber can therefore register a count while another one is in its insensitive period.

Because it cannot count at high rates without losses, the Geiger counter is now obsolete in diffractometry. It is still used in some radiation survey meters.

7-7 SCINTILLATION COUNTERS

This type of counter exploits the ability of x-rays to cause certain substances to fluoresce visible light, as in the fluorescent screens mentioned in Sec. 1-8. The amount of light emitted is proportional to the x-ray intensity and can be measured by means of a phototube. Since the amount of light emitted is small, a special kind of phototube called a *photomultiplier* has to be used in order to obtain a measurable current output.

The substance generally used to detect x-rays is a sodium iodide crystal activated with a small amount of thallium. It emits violet light under x-ray bombardment. (The details of this emission are roughly as follows. Absorbed x-rays ionize some atoms, i.e., raise some electrons from the valence to the

conduction band of NaI. These electrons then transfer some of their energy to the TI^+ ion. When the excited ion returns to its ground state, light is emitted.) The light-emitting crystal is cemented to the face of a photomultiplier tube, as indicated in Fig. 7-18, and shielded from external light by means of aluminum foil. A flash of light (scintillation) is produced in the crystal for every x-ray quantum absorbed, and this light passes into the photomultiplier tube and ejects a number of electrons from the photocathode, which is a photosensitive material generally made of a cesium-antimony intermetallic compound. (For simplicity, only one of these electrons is shown in Fig. 7-18.) The emitted electrons are then drawn to the first of several metal *dynodes*, each maintained at a potential about 100 volts more positive than the preceding one, the last one being connected to the measuring circuit. On reaching the first dynode, each electron from the photocathode knocks two electrons, say, out of the metal surface, as indicated in the drawing. These are drawn to the second dynode where each knocks out two more electrons and so on. Actually, the gain at each dynode may be 4 or 5 and there are usually at least 10 dynodes. If the gain per dynode is 5 and there are 10 dynodes, then the multiplication factor is $5^{10} = 10^7$. Thus the absorption of one x-ray quantum in the crystal results in the collection of a very large number of electrons at the final dynode, producing a pulse about as large as a Geiger pulse, i.e., of the order of volts. Furthermore, the whole process requires less than a microsecond, so that a scintillation counter can operate at rates as high as 10^5 counts per second without losses. The correct counter voltage is found by the method used for the proportional counter, by plotting counting rate vs. voltage (Fig. 7-16).

As in the proportional counter, the pulses produced in a scintillation counter have sizes proportional to the energy of the x-ray quanta absorbed. But the pulse size corresponding to a certain quantum energy is much less sharply defined, as shown in Fig. 7-19 for typical proportional and scintillation (NaI · TI) counters. As a result, it is more difficult to discriminate, with a scintillation counter, between x-ray quanta of different wavelengths (energies) on the basis of pulse size.

The efficiency of a scintillation counter approaches 100 percent over the usual range of wavelengths (Fig. 7-12), because virtually all incident quanta are absorbed, even in a relatively thin crystal.

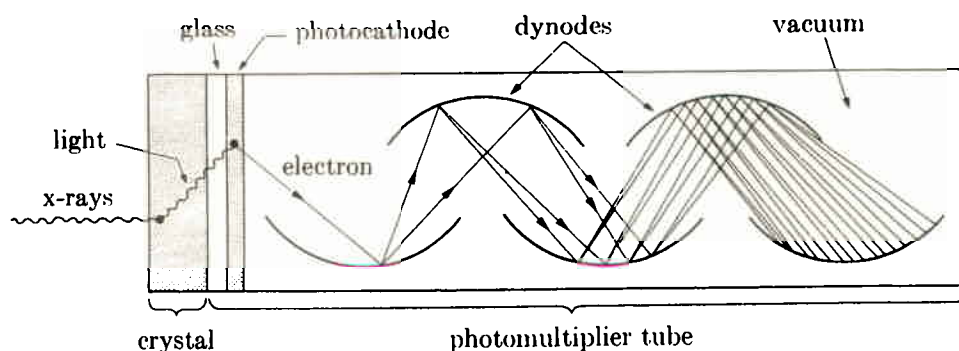


Fig. 7-18 Scintillation counter (schematic). Electrical connections not shown.

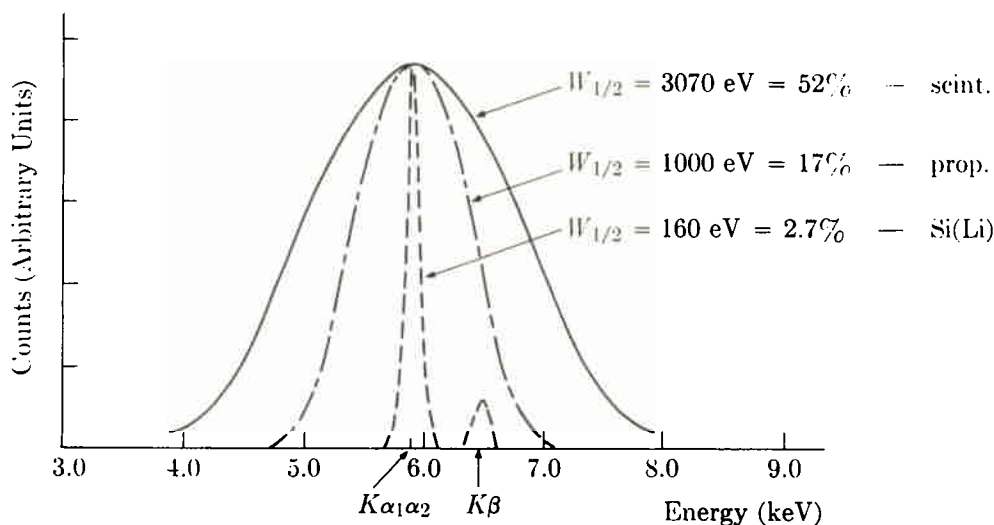


Fig. 7-19 Pulse-height distribution curves for three kinds of counter. Incident radiation is Mn $K\alpha$ ($\lambda = 2.10 \text{ \AA}$, $h\nu = 5.90 \text{ keV}$) and Mn $K\beta$ ($\lambda = 1.91 \text{ \AA}$, $h\nu = 6.50 \text{ keV}$). Frankel and Aitken [7.14].

7-8 SEMICONDUCTOR COUNTERS

Developed in the 1960s, semiconductors are the newest form of counter. They produce pulses proportional to the absorbed x-ray energy with better energy resolution than any other counter; this characteristic has made them of great importance in spectroscopy (Chap. 15). Although they have had little application in diffraction, it is convenient to describe them here along with the other counters.

Both silicon and germanium are used, germanium as a detector for gamma rays, because it is heavier and therefore a better absorber, and silicon for x-rays. Both contain a small amount of lithium, and they are designated Si(Li) and Ge(Li), inevitably referred to as "silly" and "jelly." Their properties have been reviewed by Dearnaley and Northrop [7.15], Heath [7.16], and Gedcke [7.17], among others, and in an ASTM symposium [7.18].

Pure silicon is an intrinsic semiconductor. It has very high electrical resistivity, especially at low temperatures, because few electrons are thermally excited across the energy gap into the conduction band. However, incident x-rays can cause excitation and thereby create a free electron in the conduction band and a free hole in the valence band. As shown later, the absorption of one x-ray quantum creates about a thousand electron-hole pairs. If a high voltage is maintained across opposite faces of the silicon crystal, the electrons and holes will be swept to these faces, creating a small pulse in the external circuit.

It is essential that the silicon be intrinsic (*i*). It must neither be *n*-type, containing free electrons from donor impurities, nor *p*-type, containing free holes from acceptor impurities; in either type, the free charge carriers, at their usual concentrations, would overwhelm the few carriers produced by x-rays. Production of a reasonably large intrinsic crystal, which is not easy, requires two operations:

1. The starting material is a cylindrical crystal, some 3–5 mm thick and 5–15 mm

in diameter. It is p -type, having been lightly doped with boron. Lithium is applied to one face and diffused into the crystal at an elevated temperature, producing a gradient of lithium concentration from high to low through the thickness. The lithium exists as Li^+ ions, and the free electrons it provides convert the crystal into n -type on one side, where the lithium concentration is high, leaving the other side p -type.

2. A voltage is then applied, also at an elevated temperature, to opposite faces, positive on the n side and negative on the p side (called "reverse bias"). This causes the Li^+ ions to "drift" toward the p side, resulting in a wide central region of constant lithium concentration; this region is now intrinsic because it has equal lithium and boron concentrations.

The result is the lithium-drifted silicon counter sketched in Fig. 7-20. The crystal is virtually all intrinsic, with the p and n portions confined to thin surface layers, which are exaggerated in the drawing. The very small pulses from the counter are amplified to the millivolt level by a field-effect transistor, abbreviated FET. (There is no charge amplification, such as occurs in a gas counter. The pulse from the counter contains only the charge liberated by the absorbed x-rays.)

Putting aside all of the above details of semiconductor physics, we can regard a Si(Li) counter simply as a solid-state ionization chamber, with one difference. X-rays incident on a gas ionization chamber produce a *constant* current (Sec. 7-5). In a Si(Li) counter the current flows in discrete *pulses*, because the voltage is high enough to sweep the counter free of charge carriers (the electrons and holes are highly mobile) before the next incident photon creates new carriers.

A major disadvantage of the Si(Li) counter is that it must be operated at the temperature of liquid nitrogen ($77^\circ\text{K} = -196^\circ\text{C}$) in order to minimize (1) a constant current through the detector, even in the absence of x-rays, due to thermal excitation of electrons in the intrinsic region, and (2) thermal diffusion of lithium, which would destroy the even distribution attained by drifting. Even when not

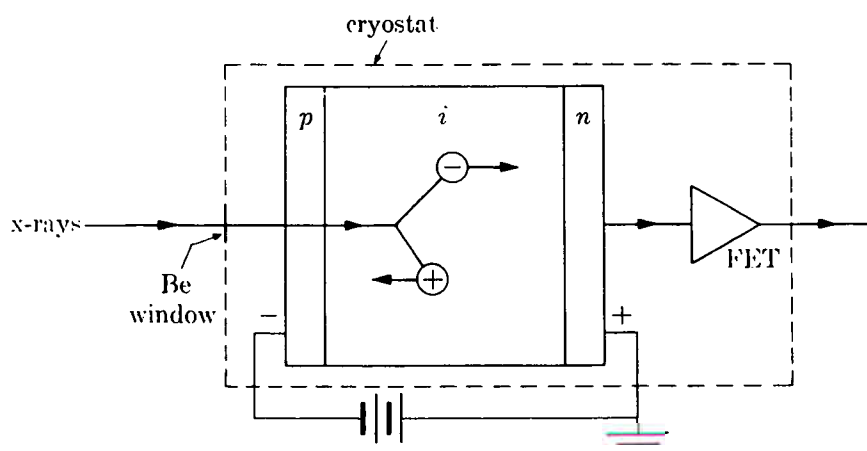


Fig. 7-20 Si(Li) counter and FET preamplifier, very schematic. Both are in a cooled evacuated space, and x-rays enter through a beryllium window. The counter is operated at about 1000 volts. \ominus = electron, \oplus = hole.

operating, the counter should not be allowed to warm too often to room temperature. Electronic "noise" in the FET increases with temperature and degrades the resolution. Thus both the counter and the FET have to be cooled, necessitating a bulky cryostat to hold several liters of liquid nitrogen (Fig. 15-10).

The efficiency of a Si(Li) counter resembles that of the other solid-state counter (scintillation), very high for intermediate wavelengths (Fig. 7-12). Very long wavelengths are partially absorbed by the counter window before they can reach the sensitive intrinsic layer. Very short wavelengths are partially transmitted by the entire counter.

The counting rate varies linearly with x-ray intensity up to rates of about 5,000-10,000 cps. Counting losses in the counter-electronics system occur in the electronics rather than the counter. The electronics are more complex than usual and include, besides the usual pulse amplifiers and shapers, a multichannel pulse-height analyzer (Sec. 7-9).

The excellent energy resolution of a Si(Li) counter is shown in Fig. 7-19. The width W of the pulse distribution is so small that the Si(Li) counter can resolve the $K\alpha$ and $K\beta$ lines of manganese, which the other two counters cannot do. Put another way, the resolution $R = W/V$ of the Si(Li) counter is 2.7 percent or some six times better than that of the proportional counter. For any kind of counter, both W and W/V vary with V , i.e., with the energy $h\nu$ of the incident x-rays. Therefore any description of counter performance must specify the x-ray energy at which it is measured; the 5.90 keV energy of the Mn $K\alpha$ line is the usual standard reference. The width W , incidentally, is often written as FWHM (full width at half maximum) in the literature of this subject.

To create an electron-hole pair in silicon at 77°K requires an average energy of 3.8 eV. The absorption of a Mn $K\alpha$ quantum should therefore create $5900/3.8 = 1550$ pairs. However, the actual number created by successive quanta might be 1540, 1560, 1555, ..., leading to a corresponding variation in the size of the output pulse. This statistical variation in the number of charge carriers created by x-ray absorption is the basic reason for the finite width W of the pulse distribution, and the same is true of proportional and scintillation counters. In the Si(Li)-FET counter, there is an even larger contribution to W , namely, electronic noise in the FET preamplifier. At the energy of Mn $K\alpha$ more than half of the observed value of W is due to noise in the FET. Beyond a certain counting rate, the resolution of the system worsens (W becomes larger) as the count rate increases.

As stated earlier, silicon counters are usually preferred for x-rays and germanium counters for the more energetic, shorter wavelength gamma rays of interest to nuclear physicists. This situation may change. Germanium counters are usable over the energy range of about 3-100 keV (4-0.1 Å), and *intrinsic* germanium crystals are now being made that do not require lithium drifting. They may find application in x-ray spectroscopy for wavelengths less than 4 Å.

In any counter except the Geiger, the average number n of ion pairs or electron-hole pairs produced is proportional to the energy E of the absorbed quantum. The actual

number has a Gaussian (normal) distribution about the mean, and the width at half maximum of this distribution is proportional to the standard deviation σ , which is equal to \sqrt{n} . Therefore the resolution R is

$$R = \frac{W}{V} = \frac{k_1 \sqrt{n}}{k_2 n} = \frac{k_3}{\sqrt{n}}, \quad (7-3)$$

where the k s are constants. The superior resolution of a Si(Li) counter is simply due to the large value of n , which is 1550 for Mn $K\alpha$. By comparison, n is only $5600/26 = 230$ for an argon proportional counter and the same radiation, because 26 eV are needed to create an ion pair in argon. Actually, the inherent resolution of a Si(Li) counter (the resolution in the absence of preamplifier noise) is even better, for complex reasons, than the above statistical argument suggests.

Because n is proportional to E , we can find from Eq. (7-3) the energy or wavelength dependence of the resolution:

$$R = \frac{k_3}{\sqrt{n}} = \frac{k_4}{\sqrt{E}} = \frac{k_4}{\sqrt{h\nu}} = k_5 \sqrt{\lambda}. \quad (7-4)$$

Although Eqs. (7-3) and (7-4) are useful for rough qualitative arguments, they do not include the substantial effect of electronic noise in Si(Li)-FET counters. A better estimate of resolution in such counters is given by

$$R = \frac{W}{V} = \frac{[(100)^2 + 2.62E]^{1/2}}{E}, \quad (7-5)$$

where E is the x-ray energy in eV and the term 100 eV in the numerator is the present level of the electronic noise. This relation is important in spectroscopy.

7-9 PULSE-HEIGHT ANALYSIS

All the counters in use today (proportional, scintillation, and semiconductor) are "proportional" in the sense that they produce pulses having a size (amplitude) that is proportional to the energy of the incident x-rays. Electrical circuits that can distinguish between pulses of different size can therefore distinguish between x-rays of different energies (wavelengths), and this ability is of great value in many experimental techniques. These circuits, in order of increasing complexity, are:

1. Pulse-height discriminator.
2. Single-channel pulse-height analyzer.
3. Multichannel pulse-height analyzer.

Circuits (1) and (2) may be used with ordinary diffractometers to increase the peak/background ratio of diffraction lines. They are by no means necessary; quite adequate diffraction patterns can be obtained from a wide variety of specimens with no other "discriminator" than a $K\beta$ filter. Circuit (3) is required only in x-ray spectroscopy (Chap. 15), in a very special kind of diffractometry (Sec. 7-10), and with a position-sensitive proportional counter. Any one of these circuits is more effective, the better the resolution of the counter with which it operates.