15 NUCLEAR COUNTING INSTRUMENTATION

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15.1 Introduction

This chapter presents descriptions of counting techniques to help the user to determine what radioanalytical measurement method(s) best suit a given need. References cited in the text provide additional details of how these measurements are made. The primary focus here is on the variables that ultimately affect the bias and precision of the counting data. The type of information that is desired—in relation to the type of radiation to detect—will determine the type of instrument and associated technique one will use to generate data. For example, samples containing a single radionuclide of high purity, sufficient energy, and ample activity may only require a simple detector system. In this case, the associated investigation techniques may offer no complications other than those related to calibration and reproducibility. At the other extreme, a sample or set of samples may require quantitative identification of many radionuclides or the laboratory may need to prepare unique calibration standards. In the latter case, specialized instruments are available. Typically, a radiochemical laboratory will encounter samples routinely that require a level of information between the two extremes described above.

A typical laboratory may be equipped with the following nuclear counting instrumentation:

- Proportional or Geiger-Mueller detectors for alpha and beta counting;
- Sodium iodide or high resolution germanium detectors for gamma detection and spectrometry;
- Solid state detectors for alpha spectrometry;
- Scintillation counters suitable for both alpha- or beta-emitting radionuclides; and
- Multichannel analyzers for alpha and gamma-ray spectrometry.

A basic requirement for accurate measurements is the use of high quality standards, traceable to a national standards organization (Section 15.9; ANSI N42.22, ANSI N42.23), to calibrate
instrumentation. Generally, with the present availability of good standards, radiochemistry laboratories rarely require instrumentation suitable for producing their own calibration standards. However, it is always advisable to compare each new standard received against the previous standard. The next three main sections of the chapter describe counting instrumentation for alpha (Section 15.2), beta (Section 15.3), and gamma (Section 15.4) radiation. In a number of cases the same instrumentation is used for radionuclides with one or more types of radiation. Note that a review covering descriptions of radionuclides, types of radiation, associated principles, and definitions for related terminology is given in Appendix A of this manual. The discussion next turns to several specific areas to cover spectrometry (Section 15.5), special instrumentation (Section 15.6), and spectrometers and energy-dependent detectors (Section 15.7). Shielding (Section 15.8) to reduce detector background of nuclear counting instruments and instrument calibration (Section 15.9) follows. This chapter closes with a discussion of other nuclear counting instrumentation considerations (Section 15.10) including a discussion on non-nuclear instrumentation (Section 15.10.4).

15.2 Alpha Counting

15.2.1 Introduction

Alpha particles are relatively massive, expend their energy over short distances, and typically exhibit limited penetration into neighboring materials. Alpha particles are also characterized by an intense loss of energy while passing through matter (see ICRU, 1992, for a discussion of dose equivalents and linear energy transfer). This loss of energy is used to differentiate alpha radioactivity from other types through the dense ionization or intense scintillation it produces. This high rate of loss of energy in passing through matter, however, also makes sample preparation conditions for alpha counting more stringent than is necessary for other types of radiation. An example of direct alpha counting to determine total alpha activity is given in ASTM C799.

Alpha radioactivity normally is measured by one of several types of detectors in combination with suitable electronic components. The detector devices most used are ionization chambers, proportional counters, silicon semiconductor detectors, and scintillation counters. The associated electronic components in all cases include high-voltage power supplies, preamplifiers, amplifiers, scalers, analog-to-digital converters, and recording devices.

The measured alpha-counting rate from a sample will depend on a number of variables. The most important of these variables are:
Geometry;  
Source diameter;  
Self-absorption;  
Absorption in air and detector window;  
Coincidence losses; and  
Backscatter.

These are discussed in detail in the literature (Blanchard et al., 1960; Hallden and Fisenne, 1963), and can be measured or corrected for in many cases by holding conditions constant during the counting of samples and standards.

Alpha counters have low backgrounds and high efficiencies. Thus, outside sources of alpha radiation will not impact the counting process and the instrument essentially focuses on the alpha source presented by the sample. However, some counters are easily contaminated internally and care should be taken to avoid contamination. Silicon detectors operated in a vacuum may become contaminated due to recoil from sources (Merritt et al., 1956). Some alpha counters are sensitive to beta radiation depending on the detector (Blanchard et al., 1960; Hallden and Fisenne, 1963). In these cases, electronic discrimination is often used to eliminate the smaller pulses due to beta particles. A discussion of alpha particle attenuation can be found in Section 15.10.1.1.

### 15.2.2 Detectors for Alpha Counting

#### 15.2.2.1 Ionization Chambers

As the incident particle enters the ionization chamber, ionization occurs through the interaction of the particle with the fill gas. The secondary electrons produced through these interactions are accelerated toward the anode as a result of the bias applied to the system. An ion current is produced at the anode as a result of the collection of the free electrons (negative ions) generated through ionization interactions. The charge collected at the anode is collected across an RC circuit resulting in a change in potential across a capacitor. The change in potential is thus related to the charge produced from the collection of electrons produced through the ionization interactions of the incident particle.

#### 15.2.2.2 Proportional Counters

As the incident particle enters the proportional counter, ionization occurs through the interaction of the particle with the fill gas. The secondary electrons produced through these interactions are accelerated toward the anode as a result of the bias applied to the system. In proportional
counters, the free electrons gain sufficient kinetic energy during acceleration to produce secondary ionization as they migrate toward the anode. This effect, known as “gas multiplication,” is used to amplify the charge collected at the anode. Similar to ionization chambers, the charge collected at the anode is collected across an RC circuit resulting in a change in potential across a capacitor. As a result of gas multiplication, the voltage pulse produced is considerably larger than the pulse produced in an ionization chamber. The magnitude of the voltage pulse is thus proportional to the original number of ion pairs formed by the incident particle.

Proportional detectors are generally constructed of stainless steel, oxygen free/high conductivity (OFHC) copper, or aluminum. No additional shielding is required for alpha proportional counting. The counter should be capable of accepting mounts up to 51 mm in diameter. Proportional counters are available in two types, either with or without a window between the sample and the counting chamber. The manufacturer’s specifications for either type should include performance estimates of background count rate, length and slope of the voltage plateau, and efficiency of counting a specified electrodeposited standard source, along with the type of gas used in the tests. For a window flow counter, the window thickness—in milligrams per square centimeter—also should be specified. With a windowless flow counter the sample and sample mount should be made of an electrical conductor in order to avoid erratic behavior due to static charge buildup.

Typical parameters for the alpha windowless flow counter are:

- background count rate = 10 counts/h or 2.8×10⁻³ cps
- length of voltage plateau = 300 V
- slope of voltage plateau = 1%/100 V for an electrodeposited source

For a window flow counter, typical values are:

- window thickness = 0.08 to 0.5 mg/cm²
- background count rate = 10 counts/h or 2.8×10⁻³ cps
- length of voltage plateau = 300 V
- slope of voltage plateau = 1%/100 V for an electrodeposited source
- efficiency = 35 to 40 percent for an electrodeposited source

15.2.2.3 Scintillation Counters

In a scintillation counter, the alpha particle transfers energy to a scintillator, such as zinc sulfide (silver activated). The transfer of energy to the scintillator results in the production of light at a
wavelength characteristic to the scintillator, and with an intensity proportional to the energy transmitted from the alpha particle. The scintillator medium is placed in close proximity to the cathode of a multiplier phototube; light photons from the scintillator strike the photo cathode, and electrons are emitted. The photoelectrons are passed through a series of dynodes resulting in the multiplication of electrons at each stage of the multiplier phototube. After amplification, a typical scintillation vent will give rise to $10^7$ to $10^{10}$ electrons, which is sufficient to serve as a signal charge for the scintillation event. The electrons are collected across an RC circuit, which results in a change in potential across a capacitor, thus giving rise to a pulse used as the electronic signal of the initial scintillation event.

The counter size is limited by the multiplier phototube size, a diameter of 51 mm being the most common. Two types of systems may be employed. In the first, the phosphor is optically coupled to the multiplier phototube and either is covered with a thin (<1 mg/cm²) opaque window or enclosed in a light-proof sample changer. With the sample placed as close as possible to the scintillator, efficiencies approaching 40 percent may be obtained. The second system employs a bare multiplier phototube housed in a light-proof assembly. The sample is mounted in contact with a disposable zinc sulfide disk and placed on the phototube for counting. This system gives efficiencies approaching 50 percent, is associated with a slightly lower background, and less chance of counter contamination.

A major advantage of alpha scintillation counting is that the sample need not be conducting. For a 51 mm multiplier phototube with the phosphor coupled to the tube, typical values obtained are a background count rate of 0.006 cps and an efficiency for an electrodeposited standard source of 35 to 40 percent. With a disposable phosphor mounted on the sample, typical values are a background count rate of 0.003 cps and an efficiency for an electrodeposited standard source of 45 to 50 percent. For both systems, voltage plateau length is 150 V with a slope of 5 percent/100 V.

15.2.2.4 Liquid Scintillation Counters

Liquid scintillation counting of alpha emitters with a commercially available instrument overcomes many of the problems inherent in other techniques (Passo and Cook 1994; Horrocks, 1974; DeFilippis, 1990; Friedlander et al., 1964; Curtis et al., 1955; Matt and Ramsden, 1964; Overman and Clark, 1960; Price, 1964; Flynn et al., 1971). Typical background counting rates range from 0.1 to 0.2 cps. Sample preparation, after radiochemical separation is performed, involves mixing the sample aliquant with a suitable liquid scintillator solution or gel phosphor before counting. In this way, planchet preparation is eliminated, volatile components are retained, and the completely enclosed sample cannot contaminate the counting chamber. Ideally, the
sample is uniformly distributed in the scintillator so there is no self-absorption. This results in a
counting efficiency of almost 100 percent. Because of the high alpha energies, considerable
chemical quenching effects can be tolerated before counting efficiency is reduced. Coincidence
losses are small in liquid scintillation counting at count rates up to $2 \times 10^4$ cps. For samples that
contain both alpha and high-energy beta emitters, difficulties do arise in distinguishing between
the two. The problem is due primarily to the broad continuum of beta energy distribution up to
the maximum energy and the poor resolution of liquid scintillation spectrometers. This problem
is aggravated because the light yield per million electron volts of alpha particles in most liquid
scintillators is approximately tenfold lower than a beta particle of equivalent energy, putting the
pulses from alphas and high-energy betas in the same region. Correction for beta activity may be
made by certain mathematical, graphical or electronic techniques (see discussion of pulse shape
discrimination in Section 15.5.4). It is preferable to separate the alpha emitter from the bulk of
the beta activity by chemistry.

15.2.2.5 Semiconductor Detectors

Semiconductor detectors used for alpha counting are essentially solid-state ionization chambers.
The ionization of the gas in an ionization chamber by alpha particles produces electron-ion pairs,
while in a semiconductor detector electron-hole pairs are produced. The liberated charge is
collected by an electric field and amplified by a charge-sensitive amplifier. In general, ion-
implanted-silicon or silicon surface barrier detectors are used for alpha counting. These detectors
are n-type base material upon which gold is evaporated to make a contact. The semiconductor
material must have a high resistivity since the background is a function of the leakage current.
This leakage current is present in an electric field since the starting material is a semiconductor,
not an insulator. The leakage current of silicon diodes doubles for every $5.5$ to $7.5$ °C change
in ambient temperature. Since the preamp HV bias resistor is a noise contributor, it is necessarily
of high value, typically 100 megohm. With a surface barrier detector having leakage current of
0.5 µA, the change in bias voltage at the detector for a $2$ °C change in ambient temperature can
be as much as 13V. This is enough bias change to affect overall gain of the detector-preamplifier
by a substantial amount. The reversed bias that is applied reduces the leakage current and a
depletion layer of free-charge carriers is created. This layer is very thin and the leakage current is
extremely low; therefore, the interactions of photons with the detector will have negligible effect.
Since the detector shows a linear response with energy, any interactions of beta particles with the
detector can be eliminated by electronic discrimination. The semiconductor is of special interest
in alpha counting where spectrometric measurements may be made since the average energy
required to produce an electron-hole pair in silicon is $3.5 \pm 0.1$ eV compared to the 25 to 30 eV
needed to produce an ion pair in a gridded ionization chamber. Consequently, silicon detectors
provide much improved resolution and also normally have lower background count rates.
The detector size is generally less than 25 mm in diameter since the resolution decreases and cost increases with detector size. For best results, the sample should be electrodeposited to make a lower mass source (Puphal and Olson, 1972). However, micro precipitation as fluorides has been reported with only slight loss of resolution (Sill and Williams, 1981; Hindman, 1983). The detector is operated in a vacuum chamber. Typical backgrounds range from $8 \times 10^{-5}$ to $2 \times 10^{-4}$ cps.

### 15.3 Beta Counting

#### 15.3.1 Introduction

This section covers the general techniques used to measure the beta particle activity resulting from radiochemical separations of specific nuclides or groups of nuclides. Beta radioactivity may be measured by several types of instruments that provide a detector and a combined amplifier, power supply, and scaler. The most widely used detectors are proportional or Geiger-Mueller counters—however, scintillation systems offer certain advantages (see discussion in Section 15.3.3). An example of the measurement of fission product activity by beta counting is given in ASTM C799, D1890, and D3648.

#### 15.3.2 Proportional Counter

Among the gas ionization-type detectors, the proportional type counter is preferable because of the shorter resolving time and greater stability of the instrument. For preparing solid sources for beta activity measurement, the sample is reduced to the minimum weight of solid material having measurable beta activity by dissolution, radiochemistry, precipitation, or ion exchange techniques. For measuring solid sources resulting from individual radiochemical separation procedures, the precipitate is appropriately mounted for counting.

Beta particles entering the sensitive region of the detector produce ionization that is converted into an electrical pulse suitable for counting. The number of pulses per unit time is directly related to the disintegration rate of the sample by an overall efficiency factor. This factor combines the effects of sample-to-detector geometry, sample self-shielding, backscatter, absorption in air and in the detector window (if any), and detector efficiency. Because most of these individual components in the overall beta-particle detection efficiency factor vary with beta energy, the situation can become complex when a mixture of beta emitters is present in the sample. The overall detection efficiency factor may be empirically determined with prepared standards of composition identical to those of the sample specimen, or an arbitrary efficiency factor can be defined in terms of a single standard such as cesium-137 ($^{137}\text{Cs}$) or other nuclide.
Gross counts can provide only a very limited amount of information and therefore should be used only for screening purposes or to indicate trends.

### 15.3.3 Liquid Scintillation

Liquid scintillation counting (LSC) avoids many sources of error associated with counting solid beta sources, such as self-absorption, back scattering, loss of activity during evaporation due to volatilization or spattering, and variable detection efficiency over a wide beta-energy range. In addition to the greatly improved accuracy offered by liquid scintillation counting, sample preparation time and counting times are significantly shorter. Sample preparation involves only adding a sample aliquant to the scintillator or gel phosphor. Because every radioactive atom is essentially surrounded by detector molecules, the probability of detection is quite high even for low-energy beta particles. Radionuclides having maximum beta energies of 200 keV or more are detected with essentially 100 percent efficiency. Liquid scintillation can, at times, be disadvantageous due to chemiluminescence, phosphorescence, quenching, or the typically higher backgrounds.

The observed count rate for a liquid scintillation sample is directly related to the beta (plus conversion electron) and positron emission rate in most cases. The important exceptions are: beta emitters whose maximum energy is below 200 keV, and counting systems wherein quenching decreases the expected photon yield, thereby decreasing the overall detection efficiency significantly below 100 percent. Low-energy beta emitters, such as tritium (³H) or carbon-14 (¹⁴C), can be measured accurately only when the appropriate detection efficiency factor has been determined with a known amount of the same radionuclide counted under identical conditions. Quenching losses are greatest at low beta energies. Quenching may be evaluated by comparison to known quench standards of the same radionuclide, using the channel ratio technique, or with other techniques as described in the manufacturer’s instructions.

For measurements in which data are expressed relative to a defined standard, the individual correction factors cancel whenever sample composition, sample weight, and counting configuration and geometry remain constant during the standardization and tests.

Liquid scintillation counting systems use an organic phosphor as the primary detector. This organic phosphor is combined with the sample in an appropriate solvent that achieves a uniform dispersion. A second organic phosphor often is included in the liquid scintillation cocktail as a wavelength shifter. The wavelength shifter efficiently absorbs the photons of the primary phosphor and re-emits them at a longer wavelength more compatible with the multiplier phototube. Liquid scintillation counting systems use either a single multiplier phototube or two...
multiplier phototubes in coincidence. The coincidence counting arrangement is less likely to accept a spurious noise pulse that occurs in a single phototube, and thus provides lower background. The requirement that both multiplier phototubes respond to each has a slight effect on the overall detection efficiency of betas with E-max >200 keV; however, system response to beta E-max <200 keV will be significant. The need to minimize detectable radioactivity in the detector and its surroundings is likewise important in liquid scintillation counting. To achieve this, scintillation-grade organic phosphors and solvents are prepared from low $^{14}$C materials such as petroleum. The counting vials are of low potassium glass or plastic to minimize counts due to potassium-40 ($^{40}$K). Liquid scintillation provides a fixed geometry from a given size counting vial and liquid volume. The calibration of liquid scintillation counting detectors is given in ASTM E181. The use of an organic phosphor for liquid scintillation counting creates a mixed waste. Chapter 20 of this manual addresses the proper disposal of these materials.

Another approach to LSC without the use of organic phosphors is Cerenkov counting. When charged particles pass through a dielectric medium, such as water, and there is an exchange of energy to the molecules of that medium, Cerenkov radiation is produced. This happens if the charged particles are moving faster than the speed of light and the exchange of energy produces electronic polarization, then when the polarized molecules return to a normal state the excess energy is released as electromagnetic radiation (Kessler, 1986). Wave shifters are usually employed to convert the ultraviolet Cerenkov radiation to the visible range. Although Cerenkov counting efficiencies are about 20 to 50 percent (Scarpitta and Fisenne, 1996) lower than when organic phosphors are used, mixed waste disposal is eliminated.

### 15.3.4 Solid Organic Scintillators

Organic scintillators, such as p-terphenyl plus a wave shifter in a plastic monomer, are polymerized to form sheet material of any desired thickness. The plastic phosphor counting system (Campion et al., 1960) has its widest use as a beta particle detector for separated, solid samples rather than for beta spectrometry applications.

The plastic beta scintillator phosphor is mounted directly on the sample and is discarded after counting. The phosphor-sample sandwich is placed in direct contact with the multiplier phototube yielding essentially a 2-π configuration. Since the output pulse of the detector system is energy dependent, the counting efficiency for a given phosphor thickness of 0.25 mm yields the highest counting efficiency with the lowest background.
Solid samples (precipitates from radiochemical separations) containing 3 to 5 mg/cm² of stable carrier are measured in such a system. For yttrium-90 (⁹⁰Y) a solid sample of this type would have a counting efficiency of 45 to 50 percent.

A plastic scintillator/phosphor system with a 25 mm multiplier phototube shielded with 12.7 mm of lead has background in the order of 4×10⁻² cps. For very low backgrounds, about 4×10⁻³ cps, the multiplier phototube and sample assembly are fitted into a well-type hollow anode Geiger tube operated in anti-coincidence. The entire assembly is then placed in a heavy shield.

The system has many advantages but reduction of background is probably most important. The reduction occurs since the scintillator does not see the surrounding mechanical components of the counter. The additional advantage of keeping the counter itself free from contamination by enclosing the phosphor-sample sandwich is also important.

A note of caution is advisable at this point. Any beta particle detection system, whether internal gas counters or scintillation counters, will detect alpha particles. It is not possible to electronically discriminate against all the alpha pulses.

If a sample is suspected of containing alpha activity, a separate alpha measurement should be made to determine the alpha contribution to the beta measurement.

15.3.5 Beta Particle Counter

The end-window Geiger-Mueller tube and the internal proportional gas-flow chambers are the two most prevalent types of detectors. Other types of detectors include scintillators and solid-state detectors. The material used in the construction of the detector and its surroundings should contain a minimal level of detectable radioactivity. If the detector is of the window-type, the window thickness may be used in calculating beta-ray attenuation; however, direct calibration of the entire counting system with standards is recommended. The manufacturer should provide all settings and data required for reliable and accurate operation of the instrument. Detectors requiring external positioning of the test sample should include a support of low-density material (aluminum or plastic), which ensures a reproducible geometry between the sample and the detector. Because different sample to detector geometries are convenient for differing sample activity levels, the sample support may provide several fixed positions ranging from 5 to 100 mm from the detector.

The detection capability for both Geiger-Mueller and proportional counters is a function of the background counting rate. Massive shielding or anti-coincidence detectors and circuitry, or both,
are generally used to reduce the background counting rate to increase the lower limit of detection (Friedlander et al., 1964). ASTM E181 covers the procedure for the calibration of beta particle counting detectors. An application of beta particle counting is given in ASTM E1005.

### 15.3.6 Associated Electronic Equipment

The high voltage power supply amplifier, scaler, and mechanical register normally are contained in a single chassis. The power supply and amplifier sections are matched with the type of detector to produce satisfactory operating characteristics and to provide sufficient range in adjustments to maintain stable conditions. The scaler should have a capacity for storing and visually displaying at least $9 \times 10^5$ counts. The instrument should have an adjustable input sensitivity matched to that of the detector, and variable high voltage power supply—an adjustable power supply and meter are unnecessary for liquid scintillation systems. Counting chambers of Geiger-Mueller and proportional counters contain a suitable counting gas and an electrode. Counting rates that exceed 200 cps should be corrected for dead time loss when using a Geiger-Mueller tube. As the applied voltage to the electrode is increased, the counting chamber exhibits responses that are characteristic of a particular voltage region. At low voltages of the order of 100 V, there is no multiplication of the ionization caused by a charged particle. At voltages approaching 1,000 V, there is appreciable amplification of any ionization within the counting chamber; however, the size of the output pulse is proportional to the amount of initial ionization. When operated in this voltage region, the device is known as a proportional counter. Usually, there is a region at least 100 V wide, known as a plateau, wherein the count rate of a standard is relatively unaffected. The operating voltage for proportional counters is selected to approximate the middle of this plateau in order to maintain stable responses during small voltage shifts. The plateau region is determined by counting a given source at voltage settings that differ by 25 or 50 V. The number of counts at each setting is recorded, and the resultant counts versus voltage are plotted. Voltage plateau curves are to be re-measured periodically to ensure continued instrument stability, or whenever an instrument malfunction is indicated. If the voltage is increased beyond the proportional region into the 1,500 to 2,000 V region, the pulse size increases and the dependence on the initial ionization intensity disappears. This is the beginning of the Geiger counting region, where a single ion pair produces the same large pulse as an intense initial ionization.

In order to eliminate alpha particle interferences a thin absorber between the sample source and the detector can be used. The absorber diameter should exceed that of the detector window. The absorber should be placed against the window to minimize beta particle scatter. Any absorber that stops alpha particles will also attenuate low energy beta particles somewhat. For example, an aluminum absorber of 7 mg/cm$^2$ will absorb 48 percent of beta particles of 350 keV maximum energy. Chemical separation of the alpha and beta particle emitters produces a higher degree of
accuracy for internal detector measurements. Published information on beta particle absorption (Friedlander et al., 1964) should be used as a guide for use of an absorber. In liquid scintillation spectra, the alpha component appears as a peak on the beta continuum and thus provides a basis for resolving the two (Bogen and Welford, 1971).

15.4 Gamma Counting

15.4.1 Introduction

This section covers the non-destructive measurement of gamma-ray radioactivity. Since gamma radiation is a penetrating form of radiation, it can be used for non-destructive measurements of samples of any form and geometry as long as standards of the same form are available and are counted in the same geometry to calibrate the detector. Because of this penetrating nature, attenuation, because of variations in sample density or sample thickness, although usually not significant, can be mathematically corrected.

When a standard cannot be obtained in the matrix and density of samples being counted, a correction for the different absorption in the matrices should be made (Modupe et al., 1993). Photons interact with matter in one of three ways: photoelectric, where all energy is transferred; Compton scattering, where only part of the energy is transferred; and pair production, where the energy creates a positron-electron pair. When the positron annihilates the electron, two 511 keV photons are emitted. Figure 15.1 shows the relative probability of each of the three predominant photon interactions with germanium.

Since different nuclides emit distinct and constant spectra of gamma radiation, the use of an energy discriminating system provides identification and measurement of all the components present in a mixture of radionuclides. General information on gamma-ray detectors and gamma counting is covered in the literature (Friedlander et al., 1964, and ICRU, 1994). Recent applications of gamma counting are given in several ASTM Test Methods (ASTM C758, C759, D3649).
Gamma counting is generally carried out using solid detectors since a gas-filled detector will not provide adequate stopping power for energetic gammas. In solids such as NaI(Tl) or CsI, the gammas interact by excitation of atoms and energy is transferred to orbital electrons and then released as light photons when the orbits are refilled. These scintillations are easily detected and amplified into useable electrical pulses by a multiplier phototube. The NaI(Tl) detector is the recommended detector for gross gamma counting because of its high efficiency and room temperature operation.

In semiconductor detectors such as Si(Li) and high-purity germanium semiconductors (HPGe), the gamma photons produce electron-hole pairs and the electrons are collected by an applied electrical field. A charge-sensitive preamplifier is used to detect the charge transferred and produce a useable electrical pulse. The semiconductor detectors are widely used in gamma spectrometry.

The output pulses from the multiplier phototube or preamplifier are directly proportional to the amount of energy deposited, which could either be total and included in the photopeak, or fractional and included in the continuum or escape peaks, in the detector by the incident photon. The pulses may be counted using a scaler or analyzed by pulse height to produce a gamma-ray spectrum.

Gamma photons interact with the detector by three distinct processes. The photoelectric effect results in complete absorption of the photon energy and produces the full energy or photopeak shown. The Compton effect results in a partial absorption of the photo energy and a scattered photon of lower energy results. The scattered photon carries energy away and the Compton continuum results (Figure 15.2). The third interaction is pair production, which occurs at energies above 1,022 keV and results in the conversion of the photon to mass as an electron-positron pair. The electron and positron give up their kinetic energy to the detector and the resulting electron joins the electron population of the detector; the positron, however, is annihilated in combining with an electron and produces two gamma.
photons of 511 keV each. One or both of the 511 keV photons may escape from the detector without interacting and the single escape and double escape peaks result.

The Comptons, from a higher energy photon, always present an interference problem in the counting of gamma photons and appropriate corrections should be made for this effect. Pair production can also be considered as an interference since the escape peaks may have an energy equal to the lower energy gamma of interest. The Compton and pair production effects can be very significant interferences and should be corrected.

The change of the absorption coefficient with gamma energy results in a wide variation of detection efficiency. The detection efficiency falls rapidly as gamma energy increases for a fixed size of detector. Two other important effects are seen as a result of the variation of the absorption coefficient; firstly, low energy photons may be absorbed in massive samples as sample thickness increases, such as large bottles of water, and erroneous results may be obtained. A similar absorption effect is seen in HPGe systems where the can around the detector acts as an absorber for very low-energy gammas and the efficiency passes through a maximum usually around 100 keV. The second result is that for low energy gammas a thin detector may be as efficient as a much thicker one since the low-energy gammas are easily stopped in the thin detector. Additionally, thin detectors will have better low energy detection limits because of reduced background interactions.

Because of this variation in efficiency and the possible interferences from other activities, gross gamma counting is only reliable when used to compare standards and samples of the same nuclide. The use of gross gamma monitoring systems should be avoided when possible and, in all cases, proper allowance should be made for the lack of accuracy.

At high count rates, random sum peaking may occur. Two absorptions may occur within the resolving time of the detector and electronics and are summed and seen as one pulse. For a detector of resolving time, t, and a count rate of A counts per unit time, the time window available for summing is 2At (since the count summed could occur as early as t before or as late as t after the other count) and the probability of another count at any time is simply A. Therefore, the sum count rate will be 2At in unit time. Random summing is strongly dependent on the count rate A and, if summing occurs, it can be reduced by increasing the sample to detector distance. Modern electronics, both conventional analog and digital (preamplifiers, amplifiers, and analog-to-digital converters) are capable of processing 100,000 cps without any significant loss of resolution. This is because of the very short time constants (resolving time) these systems are capable of producing. Over all detector performance can be affected by count rate because reduced time constants are required which will cause some loss of resolution. When a photon
interaction takes place (an event is detected), charge carriers in the form of holes and electrons are produced. The electrical field produced by the detector’s high voltage bias supply causes these carriers to be swept toward the P and N electrodes of the detector. The time it takes the carriers to travel to the electrodes is called the “charge collection time.” At very high count rates the detector continues collecting events but the data is not valid. If a second (or third) event takes place while the first set of charge carriers are still in transit, the energy from the two events get added together. Therefore, if a 2,000 keV event arrives while a 1,000 keV event is in transit, the detector would “see” a single 3,000 keV event, producing a random sum peak on pulse pileup.

When the detector starts reporting more sum peaks than valid events, you have exceeded its count rate capability. Random pulse summing or pileup can also cause peak shape and risetime problems. But the real upper limit to a detector throughput is pulse summing. This problem can be reduced or eliminated by either reducing the number of events the detector “sees” by moving the sample further away, collimate the detector, or use a smaller, less efficient detector; the smaller the detector the shorter the charge collection time, which means a higher count rate limit. Peak shifts may also occur with high count rates and short time constants. Another factor that will affect high count rate performance is improper setting of the amplifier pole zero. Improper setting of the pole zero with either under or over shooting of input pulse will effect peak resolution.

Well counters that have very high efficiencies are prone to summing since, for a given source strength, the count rate is higher than for a detector of lower efficiency. For moderate and high-source strengths, the trade-off is a poor one and the well counter is best suited for low-level work where its high efficiency is an important advantage.

Cascade summing may occur when nuclides that decay by a gamma cascade are counted. Cobalt-60 ($^{60}$Co) is an example; 1,173.2 keV and 1,332.5 keV from the same decay may enter the detector and be absorbed, giving a 2,505.7 keV sum peak. Another example of Cascade summing occurs when counting sodium-22 ($^{22}$Na) close to the detector (see Figure 15.3). Cascade summing may be reduced and eventually eliminated by increasing the source-to-detector distance.

**FIGURE 15.3 Energy Spectrum of $^{22}$Na**
The resolution of a gamma detector is the effective limit to its utility even when complex data reduction methods are used. A typical 76x76 mm NaI(Tl) detector will give full-width half-maximum (FWHM) of approximately 60 keV at 661.6 keV gamma energy and approximately 90 keV at 1,332.5 keV gamma energy.

15.4.2 Energy Efficiency Relationship

Because of the rapid falloff in gamma absorption as gamma energy rises, the detection efficiency shows a similar effect. Figure 15.4 shows a typical efficiency vs. energy plot of a 70 percent HPGe p-type, a 35 percent HPGe n-type, and HPGe well detectors of 122 cm³ with a vespel well and 320 cm³ with a Mg well. The portion of the curve for n-type and well detectors at low energies shows that as the absorption coefficient increases geometry becomes the limiting factor. The maximum efficiency for both co-axial detectors is well below 50 percent due to the presence of a beta absorber, the containment of the detector and the geometry effect. The p-type detector shows significant low energy efficiency drop off because of the absorption of gamma rays in the detector’s inactive Ge dead layer. The well detector shows excellent efficiency below 100 keV because of the geometry effect and absence of an attenuating germanium dead layer. The 76x76 mm NaI(Tl) detector is the most widely used size. A large amount of data are available in the open literature on both the use and results obtained with detectors of this size. Heath (1964) has written a comprehensive review and supplied many gamma-ray spectra in both graphical and digital form.

Other sizes of detectors may be used. However, the following should be noted: smaller detectors, such as 38x38 mm, will give efficiencies that are low and fall off more rapidly as gamma energy increases. Small or thin detectors are useful for the measurement of low-energy gammas since they are less responsive to high-energy gammas and the interference from Compton effects is reduced. This will result in a lower background.
Larger detectors will give higher efficiencies and less falloff as gamma energy increases. Larger detectors are useful for situations where the highest attainable efficiency is desired and for the assembly of complete absorption detectors. The increase in efficiency is accompanied by an increased background count rate and an increase in the probability of summing in the detector.

Well detectors will give very high efficiencies, up to about 80 percent for low and moderate energy gammas. The well detector is useful for low levels of activity and the background of a well detector is essentially the same as that of a plain cylindrical detector of the same overall dimensions. Summing becomes a definite problem at high activities since both random and cascade summing result from the high efficiencies and the high geometry of the well detector.

Detector efficiency will also vary as a function of sample geometry. Table 15.1 gives counting efficiencies obtained with various sample geometries for a 55 percent HPGe detector.

**TABLE 15.1 Typical Percent Gamma-ray Efficiencies for a 55 Percent High-Purity Germanium Detector with Various Counting Geometries**

<table>
<thead>
<tr>
<th>ENERGY (keV)</th>
<th>FILTER PAPER</th>
<th>50 cm³ PLANCHET</th>
<th>90 cm³ AL CAN</th>
<th>600 cm³ MARINELLI BEAKER</th>
</tr>
</thead>
<tbody>
<tr>
<td>60</td>
<td>15.6</td>
<td>14.6</td>
<td>11.6</td>
<td>5.0</td>
</tr>
<tr>
<td>88</td>
<td>15.2</td>
<td>14.2</td>
<td>11.3</td>
<td>7.4</td>
</tr>
<tr>
<td>122</td>
<td>15.1</td>
<td>12.6</td>
<td>10.2</td>
<td>8.4</td>
</tr>
<tr>
<td>166</td>
<td>12.0</td>
<td>9.6</td>
<td>8.0</td>
<td>7.9</td>
</tr>
<tr>
<td>279</td>
<td>9.3</td>
<td>7.4</td>
<td>6.0</td>
<td>6.1</td>
</tr>
<tr>
<td>392</td>
<td>7.2</td>
<td>5.5</td>
<td>4.5</td>
<td>4.8</td>
</tr>
<tr>
<td>514</td>
<td>5.4</td>
<td>4.2</td>
<td>3.5</td>
<td>3.8</td>
</tr>
<tr>
<td>662</td>
<td>4.7</td>
<td>3.6</td>
<td>3.0</td>
<td>3.1</td>
</tr>
<tr>
<td>835</td>
<td>3.9</td>
<td>2.9</td>
<td>2.4</td>
<td>2.7</td>
</tr>
<tr>
<td>898</td>
<td>3.1</td>
<td>2.4</td>
<td>2.1</td>
<td>2.2</td>
</tr>
<tr>
<td>1115</td>
<td>3.0</td>
<td>2.3</td>
<td>1.9</td>
<td>2.1</td>
</tr>
<tr>
<td>1173</td>
<td>2.6</td>
<td>2.0</td>
<td>1.7</td>
<td>1.8</td>
</tr>
<tr>
<td>1333</td>
<td>2.3</td>
<td>1.8</td>
<td>1.5</td>
<td>1.6</td>
</tr>
<tr>
<td>1836</td>
<td>1.7</td>
<td>1.3</td>
<td>1.2</td>
<td>1.3</td>
</tr>
</tbody>
</table>

*Although the counting efficiencies listed above were obtained with a 55 percent (relative to a 3x3 inch NaI detector) HPGe detector, the calculation of counting efficiencies by extrapolation for detectors with different relative efficiencies is not possible. This is because detectors with the same relative efficiency may be of significantly different dimensions thus producing a detector/sample solid angle very different than what was used to prepare this table.*
15.4.3 Sodium Iodide Detector Assembly

A cylindrical 76x76 mm NaI detector is activated with about 0.1 percent thallium iodide, with or without an inner sample well, optically coupled to a multiplier phototube, and hermetically sealed in a light-tight container. The NaI(Tl) crystal should contain less than 5 ppm of potassium and be free of other radioactive materials. In order to establish freedom from radioactive materials, the manufacturer should supply a gamma spectrum of the background of the detector between 0.08 and 3,000 keV. The resolution of the detector for the 662 keV gamma from $^{137}$Cs decay should be less than 50 keV FWHM or less than 7 percent when measured with the source in contact with the end cap.

The following components are required for a complete NaI(Tl) gamma-ray spectrometry system:

- **High-Voltage Power Supply**: 500 to 2,000 V dc regulated to 0.1 percent with a ripple of not more than 0.01 percent
- **Preamplifier**: Linear amplifier system to amplify the output from the multiplier phototube to a maximum output of 10 V.
- **Analyzer with Scaler and Timer**: A single-channel discrimination system will accept all or any part of the output from the amplifier and pass it to the scaler. Any pulses lying outside the preset limits are rejected. The lower limit is usually referred to as the *threshold* and the difference between the two limits is the *window*.

Sample mounts and containers may consist of any reproducible geometry container that is commercially available. Other considerations are cost, ease of use, disposal, and effective containment of radioactivity for the protection of the workplace and personnel from contamination.

- **Beta Absorber**: A beta absorber of 3 to 6 mm of aluminum, beryllium, or poly(methyl methacrylate) should completely cover the upper face of the detector to prevent betas from reaching the detector.
15.4.4 High Resolution Germanium Detectors

High resolution germanium detectors are produced from very high purity material, the required level of impurities in the detector crystal is usually less than $10^9$ atom/cm$^3$. Any type of germanium—either planar, co-axial or well-configuration—cannot be operated at room temperature because of the large thermally induced leakage current that results. These detectors should be cooled in order to reduce the thermal generation of charge carriers (thus reverse leakage current) to an acceptable level. Otherwise, leakage current induced noise reduces the energy resolution of the detector. The detector is mounted in a vacuum chamber which is attached to or inserted into an liquid nitrogen (LN2) dewar or an electrically powered cooler. The sensitive detector surfaces are thus protected from moisture and condensation contaminants.

The boiling point of liquid nitrogen (77 °K) is usually taken advantage of to reduce the operating temperature of the detector. Since germanium detectors can be operated at temperatures as high as 130 °K, mechanical closed-cycle refrigerators can also be used. These systems can cool a detector to as low as 50 °K. Therefore, with proper thermal control the detector can be cooled to its optimum operating temperature. The required preamplifier is normally included as part of the cryostat. In this configuration the preamplifier can also be cooled to reduce electronic noise.

HPGe detectors are preferred for the analysis of complex gamma-ray spectra involving many nuclides and peaks. However, for samples with only a few nuclides, the complexity of an HPGe system may not be cost effective. The calibration of germanium detectors is given in ASTM E181.

15.4.5 Low Background High Resolution Germanium Detectors

Environmental samples requiring the lowest possible minimum detection analyses (MDAs) should be counted with large high efficiency germanium detectors in low background cryostats. Most of the background from naturally occurring radionuclides such as $^{40}$K from building materials, radon decay products, and cosmic rays can be reduced by proper shielding. However, naturally occurring $^{235}$U, $^{238}$U, $^{232}$Th, and anthropogenic $^{137}$Cs and $^{60}$Co may be present in cryostat materials. With careful selection and substitution of materials, low background gamma-ray systems can be fabricated. Germanium crystal mountings and detector end caps have been fabricated with magnesium to eliminate aluminum contaminated with radioactive thorium isotopes. Figures 15.5 and 15.6 show shielded background spectra obtained with 56 percent germanium detectors in standard and extra low background cryostats.
15.4.6 High Resolution Detectors for Low Energy Spectrometry

High resolution low gamma-ray energy detectors are available in various configurations. The commonly used ones are either high purity germanium or silicon. The various detector types include: planar (Ge or Si), low-energy germanium (LEGe), reverse-electrode germanium (REGe) and extended-range germanium (XtGe). These detectors are equipped with beryllium entrance windows to reduce attenuation. These detectors are especially useful for measuring nuclides that emit gamma or X-rays from a few keV to about 150 keV.

15.4.7 CsI(Tl) Detectors

CsI(Tl) crystals have the highest light output of all known scintillators. However, because light output is not well matched to the sensitivity of the photocathode of a multiplier phototube, the yield for gamma rays is only 45 percent of the efficiency of NaI(Tl). With the proper electronics, CsI(Tl) detectors can be used for α-particle energy discrimination.

15.4.8 CdZnTe Detectors

These gamma-ray detectors, in addition to only being produced in very small volumes, do not have energy resolutions as good as HPGe but are better than NaI(Tl). Their greatest advantage is...
their ability to operate at room temperature. Because of their small size and resulting low gamma-ray detection efficiency, they are useful for the analysis of very high level sources.

15.4.9 BGO Detectors

Because bismuth germanate (Bi$_4$Ge$_3$O$_{12}$) is a high Z, high density (7.13 g/cm$^3$), scintillation material, it is a very efficient gamma-ray absorber. Although BGO crystals have very good peak-to-Compton ratio, their effective efficiency is only 10 to 15 percent as good as a NaI(Tl) crystal. However, BGO is a relatively hard, rugged, non-hygrosopic crystal which does not cleave or absorb any significant amount of the scintillation light. The crystal housing does not require hermetic air-tight sealing. These crystals are useful in applications where high photofraction is required.

15.5 Spectrometry Systems

This section will present a number of different type of detector systems commonly use for gamma-ray spectrometry.

15.5.1 Alpha/Gamma Coincidence Systems

Alpha/Gamma Coincidence Systems have been used for the direct measurement of $^{224}$Ra and $^{226}$Ra. The counting technique is based upon the coincidence measurement of the characteristic particle-photon emissions of these isotopes. Silver activated zinc sulfide for alpha detection is combined with a NaI well for gamma-ray detection (McCurdy, 1981).

15.5.2 Beta/Gamma Coincidence Systems

Many radionuclides remain in an excited state after what may be considered beta decay. This results in the emission of a gamma ray as the decay process goes to the ground state. A beta/gamma coincidence system will have significantly improved lower limit of detection over a beta or a gamma counting system because of its very low background. Systems have been designed with both 2-$\pi$ and 4-$\pi$ geometry (McCurdy et al., 1980).

15.5.3 Gamma/Gamma Coincidence Systems

These counting systems can provide extremely low backgrounds and are very useful for analyzing those radionuclides that decay with cascading (coincident) gamma rays. The systems usually consist of two large NaI(Tl) detectors with a surrounding active anti-coincidence shield.
of either NaI(Tl) or plastic phosphor. However, HPGe detectors have also been used in place of
the two large NaI(Tl) detectors. Only gamma-ray pulses that are detected in both of the primary
detectors at the same time (coincident) and not in the active shield are recorded. Even though
these systems can be large, because of the shielding requirements for two detectors and an active
annulus, and require complex electronics, the improvement in lower limit of detection for certain
radionuclides is worth the investment (Perkins, 1965; Sanderson, 1969).

15.5.4 Photon-Electron Rejecting Alpha Liquid Scintillation Systems

Another technique for the analysis of alpha emitting radionuclides combines liquid scintillation
counting with pulse shape discrimination to significantly reduce background counts from photo-
electrons produced by ambient background gamma rays and to eliminate interferences from beta
emitters in the sample/scintillation cocktail. Pulse shape discrimination electronically selects only
pulses produced by alpha particles because of their longer decay times in the scintillation
solution. Typical alpha peak resolutions are about 5 percent. Typical detectable activities for
alpha emitters such as $^{234}$U and $^{241}$Am are 0.0037 and 0.37 Bq (0.1 and 10 pCi).

15.6 Special Instruments

This section covers some radiation detection instruments and auxiliary equipment that may be
required for special application in the measurement of radioactivity.

15.6.1 4-π Counter

The 4-π counter is a detector designed for the measurement of the absolute disintegration rate of
a radioactive source by counting the source under conditions that approach a geometry of 4-π
steradian. Its most prevalent use is for the absolute measurement of beta emitters. For this
purpose, a gas-flow proportional counter is commonly used. 4-π counting systems consist of two
hemispherical or cylindrical chambers whose walls form the cathode, and a looped wire anode in
each chamber. The source is mounted on a thin supporting film between the two halves, and the
counts recorded in each half are summed.

Gamma-ray and hard X-ray counters with geometries approaching 4-π steradian can be
constructed from both NaI(Tl) or germanium crystals in either of two ways. A well crystal (that
is, a cylindrical crystal with a small axial hole covered with a second crystal) will provide nearly
4-π geometry for small sources, as will two solid crystals placed very close together with a small
source between them. The counts from both crystals are summed as in the gas-flow counter. The
deviation from 4-π geometry can be calculated from the physical dimensions. For absolute
gamma-ray counting, the efficiency of the crystal for the gamma energy being measured and the
absorption in the detector end cap should be taken into account. The liquid scintillation counter is
also essentially a 4-π counter for alpha and beta particles, since nearly all the radiations are
emitted into and interact with the detecting medium.

15.6.2 Low-Geometry Counters

This type of instrument is particularly useful for the absolute counting of alpha particles. The
alpha emitter, in the form of a very thin solid source, is placed at a distance from the detector
such that only a small fraction (<1 percent) of the alpha particles are emitted in a direction to
enter the counter. This solid angle is obtained from the physical measurements of the instrument.
The space between the source and the detector is evacuated to eliminate the loss of alpha
particles by absorption in air. The detector can be any counter that is 100 percent efficient for all
alpha particles that enter the sensitive volume—a gas-flow proportional counter with a window
that is thin (approximately 1 mg/cm²) compared to the range of the alpha particles or the
semiconductor alpha detector with a 1 mg/cm² covering. The advantages of this instrument for
absolute alpha counting are that the effect of absorption of alpha particles in the source itself is
kept to a minimum since only particles that travel the minimum distance in the source enter the
detector (particles that have longer paths in the source are emitted at the wrong angle), and back-
scattered alpha particles (those that are emitted into the source backing and are reflected back up
through the source) lose sufficient energy so that they cannot enter the detector. One such
instrument is described in Curtis et al. (1955).

15.6.3 Internal Gas Counters

The internal gas counter is so named because the radioactive material, in the gaseous state, is
placed inside a counting chamber and thus becomes part of the counting gas itself. It is useful for
high-efficiency counting of weak beta- and X-ray emitting radionuclides. The radiations do not
have to penetrate a counter window or solid source before entering the sensitive volume of a
detector. The counter may be an ionization chamber, or it may be operated in the Geiger or
proportional mode. Most present-day instruments are of the latter type, and they generally take
the form of a metal or metal-coated glass cylinder as a cathode with a thin anode wire running
coaxially through it and insulated from the cylinder ends. A wire through the wall makes
electrical contact to the cathode. The counter has a tube opening through which it may be
connected to a gas-handling system for filling. The purity of the gas is important for efficient and
reproducible counting, particularly in the proportional mode.
In a modification of the internal gas counter, scintillation counting has been used. The inner walls of the chamber are coated with a scintillation material and the radioactive gas is introduced. An optical window is made a part of the chamber, and the counting is done by placing this window on a multiplier phototube to detect the scintillations. This system is particularly useful for counting radon gas with zinc sulfide as the scintillator. Additional details on internal gas counting may be found in Watt and Ramsden (1964).

15.7 Spectrometers and Energy-Dependent Detectors

The availability of energy-dependent detectors (detectors whose output signal is proportional to the energy of the radiation detected) that are easy to operate and maintain and have good resolution makes it possible to measure not only the total activity of a radioactive sample but the energy spectrum of the nuclear radiations emitted. Nuclear spectrometry is most useful for alpha particles, electromagnetic radiation (gamma and X-rays), and conversion electrons, since these radiations are emitted with discrete energies. Beta spectra have more limited use since beta particles are emitted from a nucleus with a continuous energy distribution up to a characteristic maximum (E- max), making a spectrum containing several different beta emitters difficult to resolve into its components. The advantages of spectrometric over total activity measurements of radioactive sources are increased selectivity, detection limit, and accuracy because nuclide identification is more certain, interference from other radioactive nuclides in the sample is diminished or eliminated, and counter backgrounds are reduced since only a small portion of the total energy region is used for each radiation.

The detectors for alpha spectra are gridded ion-chambers and silicon semiconductor detectors. Gridded ion-chambers are no longer available commercially and should be constructed by the user. A variety of semiconductor detectors for alpha spectrometry are commercially available. These detectors have essentially replaced ion-chambers, although the chambers have the advantages of high efficiency (nearly 50 percent) for large-area sources.

Silicon alpha particle detectors have a depletion region which is formed by applying a high voltage bias. The electric field produced collects the electron-hole pairs produced by incident alpha particles. Either surface barrier or passivated ion-implanted silicon are commonly used for spectrometry.

The principal detectors used for gamma-ray spectrometry are thallium-activated sodium iodide scintillation crystals, NaI(Tl), and high purity germanium semiconductors, HPGe. HPGe detectors are available in n-type and p-type germanium. P-type germanium detectors have dead layers which produce entrance windows from 500 to 1,000 µm thick. On the other hand, n-type
detectors have extremely thin entrance windows of about 0.3 μm. These n-type detectors when housed in an end cap with a beryllium window are excellent for measuring both low energy and high energy (3 to 10,000 keV) gamma rays. However, applications which require the best possible energy resolution, peak shape, and efficiency for gamma-ray measurements above 80 keV, p-type HPGe is the detector material of choice.

For X-rays and very low-energy gamma rays, lithium-drifted silicon semiconductor Si(Li), planar germanium, and gas-filled thin window (approximately 1 mg/cm²) proportional counters are used.

The electronic version of Heath’s (1964) Ge(Li) and Si(Li) Detector Gamma-ray Spectrum Catalogue is available in two forms. The document is on the Web at http://id.inel.gov/gamma; it is also available on a CD-ROM.

A complete NaI(Tl) detector spectrometer requires a high-voltage power supply for the phototube (usually operated at 600 to 1,000 V), a preamplifier, linear amplifier, pulse-height analyzer, and output recorder. Because NaI(Tl) detectors cannot resolve gamma-ray energies that are only a few keV apart, a least-squares computer program should be used to quantify a complex gamma-ray spectrum.

Germanium and silicon detectors are junction-type semiconductor devices. With silicon detectors, a sensitive region is produced by drifting lithium under the influence of an electric field at an elevated temperature (100 to 400 °C) into the crystal. The crystal then functions as a solid ion chamber when a high voltage is applied. Today, germanium detectors are made with very high purity material that does not require lithium drifting. In order to obtain high resolution, these detectors should be operated at low temperatures to reduce thermal noise. At room temperature, sufficient free electrons will be present in the crystal to obscure the measurement of gamma and X-rays. Consequently, the detectors are operated at liquid nitrogen temperatures by a cryostat consisting of a metallic cold-finger immersed in a Dewar flask containing liquid nitrogen or mechanically refrigerated.
The electronic components required to obtain spectra are similar to those for NaI(Tl) detectors, except that because smaller pulses should be measured, high-quality electronics should be used. A complete HPGe system includes a high-voltage bias supply for the detector, a preamplifier, amplifier (usually charge-sensitive), pulse height analyzer, and recording device. With the exception of extremely complex spectra, most high resolution spectra can be quantified by simple integration of full energy gamma-ray peaks.

The resolution of gamma-ray detectors is usually specified in terms of its FWHM. Detector resolution, expressed in percent, improves with increasing energy and for NaI(Tl) detectors and is usually determined from the 662 keV gamma ray emitted in the decay of $^{137}\text{Cs}$. This is shown graphically in the gamma-ray spectrum in Figure 15.7. For HPGe detectors, $^{60}\text{Co}$ is measured 25 cm above the detector end cap. Quality sodium iodide crystals have resolutions in the range of 6.5 to 7 percent for $^{137}\text{Cs}$. Detection efficiency for the same geometry and window thickness is a function of several parameters and much published information on efficiencies for various energies, detector sizes, source-to-detector distances, and other variables is available (Crouthamel et al., 1970). The efficiency for gamma-ray detection is expressed as full energy peak efficiency; the fraction of incident gamma rays that give a full-energy peak for a particular source-detector configuration. For a 102 mm thick NaI(Tl) crystal, with the source on the surface (zero distance), this fraction is approximately 0.24 for the 661.6 keV gamma ray of $^{137}\text{Cs}$ and approximately 0.14 for the 1,332.5 keV gamma ray of $^{60}\text{Co}$. The peak-to-valley or peak-to-Compton ratio is the ratio of counts at the maximum height of the full-energy peak to the counts at the minimum of the Compton continuum. A high ratio indicates narrow peaks, that is, good resolution, for that particular energy.

The efficiency specification of a HPGe detector is expressed by comparing its $^{60}\text{Co}$, 1,332.5 keV efficiency at 25 cm with that of a 76x76 mm cylindrical NaI(Tl) detector at the same distance.
Photopeaks are spread over a much smaller energy range in germanium than in sodium iodide, the background under the peak is much less (Figure 15.8). This means that for small sources of moderately energetic gamma rays, germanium is more sensitive than sodium iodide.

Typical specifications for a germanium gamma-ray detector could include but should not be limited to the following:

**DETECTOR:** The gamma-ray detector should consist of High-Purity n-type germanium.

**SIZE:** The germanium crystal should be at least 5.5 cm in diameter and at least 7.0 cm long.

**EFFICIENCY:** The relative counting efficiency compared to a 3”x3” NaI detector at 25 cm for $^{60}$Co (1,332 keV) should be equal to or better than 50 percent.

**RESOLUTION:** The resolution (FWHM) of the detector should be equal to or better than 2.2 keV at 1,333 keV ($^{60}$Co). The resolution (FWHM) at 122 keV ($^{57}$Co) of the detector should be equal to or better than 1.0 keV. The detector resolution at FWTM should be equal to or better than 2 times the FWHM.

**PEAK-TO-COMPTON RATIO:** The peak-to-Compton ratio for 1,333 keV ($^{60}$Co) should be equal to or better than 50:1.

**BACKGROUND:** Low radioactivity materials should be used so that any full energy gamma-ray line (excluding 511 keV and 1,460 keV) present in a 1,000-minute background spectrum (100-2,000 keV) obtained in a graded 10 cm lead shield should not exceed 0.20 counts per minutes.
CONTACTS: The internal detector contacts should be DC-coupled ion implants so that low energy gamma-ray attenuation is avoided.

PREAMP: A low-noise, cooled field-effect transistor preamplifier should be used to provide the detector output signals.

CRYOSTAT: The cryostat should be constructed of low radioactivity materials throughout and should contain sufficient lead shielding in order to minimize radiation from the dewar or lower portion of the cryostat.

END CAP: The end cap should consist of a 20 mil beryllium window with 0.5 mm aluminum side walls and be no greater than 7.6 cm diameter (OD). This diameter should be maintained for at least 8 cm from the end cap. Below this point the outside diameter of the end cap may be increased. The top of the end cap should be between 95 and 102 cm above the outside base of the dewar.

TEMPERATURE: The cryostat should contain a temperature sensing circuit to provide high voltage shut down in order to prevent preamplifier damage in case of warm-up due to loss of liquid nitrogen.

Spectra of beta particles and conversion electrons can be obtained with sodium iodide and n-type HPGe detectors. A germanium detector with a volume of 120 cm$^3$ has an efficiency approximately 20 percent that of a 76x76 mm NaI(Tl) crystal. Larger HPGe detectors are available with relative efficiencies over 150 percent when compared with a 76x76 mm NaI(Tl) crystal.

Presently available germanium detectors have resolutions of 1.5 to 2.5 keV at 1,332.5 keV. The method used to measure the energy resolution is described in ANSI/IEEE 325. This greater resolution makes this detector the one of choice for gamma-ray spectrometry and cancels to some extent the higher efficiency available from sodium iodide. Since the pulses from a single semiconductor detectors sufficiently thick (a few centimeters) to absorb the particles completely. One disadvantage of sodium iodide detectors is their relatively thick entrance windows. Other semiconductor detectors have thin beryllium entrance windows and can be used for beta spectrometry.

Good spectra of low-energy beta particles, conversion electrons, and X-rays can be obtained with a gas-flow proportional Counter provided that a linear preamplifier is used. The resolution is intermediate between NaI(Tl) and HPGe. Organic scintillators, such as anthracene and polystyrene polymerized with scintillating compounds, are also useful for beta spectrometry.
They are packaged with a phototube in a manner similar to sodium iodide crystals. Liquid scintillation mixtures also give beta spectra, and the output of a commercial liquid scintillation counter is usually fed into a multichannel pulse-height analyzer to obtain a beta energy spectrum (Blanchard et al., 1960). A spectrum of $^{210}\text{Pb}$, $^{210}\text{Bi}$, and $^{210}\text{Po}$ in Figure 15.9 shows the resolution obtainable by liquid scintillation counting of aqueous samples in a dioxane-based solution. The $^{210}\text{Bi}$ curve is from a beta particle, and the $^{210}\text{Po}$ peak is from an alpha particle. Organic scintillators are preferable to sodium iodide for beta spectrometry because less back scattering occurs.

15.7.1 Anti-Coincidence Counters

Substantial background reduction can be achieved in beta and gamma counters by surrounding or covering the sample detector with another detector also sensitive to beta or gamma radiation, and connecting them electronically so that any pulse appearing in both detectors at the same time is canceled and not recorded as a count. This is referred to as anti-coincidence shielding, and is recommended for obtaining very low backgrounds. This type of counter was used for many years in directional studies of cosmic rays, and was first applied to reducing the background of beta counters by Libby in his study of natural $^{14}\text{C}$. The thick metal shielding (lead, iron, or mercury) ordinarily used to reduce cosmic-ray and gamma-ray background should also be present, and is placed outside the anti-coincidence shielding.

Anti-coincidence shielding of gamma-ray detectors operates in a similar way, and is particularly useful in reducing the Compton continuum background of gamma rays (Nielson, 1972). Gamma rays that undergo Compton scattering and produce a pulse in both the detector and the anti-coincidence shield are canceled electronically. Ideally, only those gamma rays that are completely absorbed in the sample detector produce a count that is recorded with the total energy of the gamma ray (full-energy peak). There are second-order effects that prevent complete elimination.
of Compton scattering, but the improvement is substantial (Perkins, 1965, and Cooper et al., 1968).

### 15.7.2 Coincidence Counters

In coincidence counting, two or more radiation detectors are used together to measure the same sample, and only those nuclear events or counts that occur simultaneously in all detectors are recorded. The coincidence counting technique finds considerable application in studying radioactive decay schemes; but in the measurement of radioactivity, the principal uses are for the standardization of radioactive sources and for counter background reduction.

Coincidence counting is a very powerful method for absolute disintegration rate measurement (Friedlander et al., 1964; IAEA, 1959). Both alpha and beta emitters can be standardized if their decay schemes are such that $\beta-\gamma$, $\gamma-\gamma$, $\beta-\alpha$, or $\alpha-X$-ray coincidence occur in their decay.

Gamma-gamma coincidence counting with the source placed between two sodium iodide crystals, is an excellent method of reducing the background from Compton scattered events. Its use is limited, of course, to counting nuclides that emit two photons in cascade (which are essentially simultaneous), either directly as in $^{60}$Co, by annihilation of positrons as in $^{65}$Zn, or by immediate emission of a gamma ray following electron capture decay. Non-coincident pulses of any energy in either one of the crystals will be canceled, including cosmic-ray photons in the background and degraded or Compton scattered photons from higher energy gamma rays in the sample. Thus, the method reduces interference from other gamma emitters in the sample. When two multichannel analyzers are used to record the complete spectrum from each crystal, singly and in coincidence, then the complete coincident gamma-ray spectrum can be obtained with one measurement. The efficiency for coincidence counting is low since it is the product of the individual efficiencies in each crystal, but the detection limit is generally improved because of the large background reduction (Nielsen and Kornberg, 1965). This technique is often referred to as two-parameter or multidimensional gamma-ray spectrometry.

Additional background improvement is obtained if the two crystals are surrounded by a large annular sodium iodide or plastic scintillation crystal connected in anti-coincidence with the two inner crystals. In this case a gamma ray that gives a pulse, but is not completely absorbed in one of the two inner crystals, and also gives a pulse in the surrounding crystal, is canceled electronically (Perkins, 1965, and Nielsen and Kornberg, 1965). This provides additional reduction in the Compton scattering background. Germanium detectors may be used in place of the inner sodium iodide crystals for improved resolution and sensitivities (Cooper et al., 1968).

An example of an assay for plutonium content using passive thermal-neutron coincidence
counting is given in ASTM (C1207). Another example of passive thermal-neutron coincidence counting using a moveable californium source is given in ASTM (C1316).

15.8 Shielding

The purpose of shielding is to reduce the background count rate of a measurement system. Shielding reduces background by absorbing some of the components of cosmic radiation and some of the radiations emitted from material in the surroundings. Ideally, the material used for shielding should itself be free of any radioactive material that might contribute to the background. In practice, this is difficult to achieve as most construction materials contain at least some naturally radioactive species (such as $^{40}$K, members of the uranium and thorium series, etc.). The thickness of the shielding material should be such that it will absorb most of the soft components of cosmic radiation. This will reduce cosmic-ray background by approximately 25 percent. Cosmic-ray interactions in lead shields will produce lead X-rays that are in turn shielded by cadmium and copper liners. Such a shield is referred to as a “graded shield.” Six millimeters of oxygen-free high-conductivity (OFHC) copper can also be used to reduce the cosmic-ray produced lead X-rays without the cadmium liner. Shielding of beta- or gamma-ray detectors with anti-coincidence systems can further reduce the cosmic-ray or Compton scattering background for very low-level counting.

Detectors have a certain background counting rate from naturally occurring radionuclides and cosmic radiation from the surroundings; and from the radioactivity in the detector itself. The background counting rate will depend on the amounts of these types of radiation and on the sensitivity of the detector to the radiations.

In alpha counting, low backgrounds are readily achieved since the short range of alpha particles in most materials makes effective shielding easy. Furthermore, alpha detectors are quite insensitive to the electromagnetic components of cosmic and other environmental radiation.

The size and interior dimensions of shields constructed for gamma-ray spectrometry or gamma counting in general should be considered so that sample back scatter radiation from the shield wall to the detector is minimized. In general, shield wall should be at least 10 cm from the detector. Back scatter radiation will fall off as the square of the detector to shield wall distance.

15.9 Instrument Calibration

Calibrations of instruments should be made using reference materials of known and documented value and stated uncertainty. These reference materials should be supplied by:
National Institute of Science and Technology (NIST) directly;

A standard source supplier whose measurement capabilities and/or manufacturing processes are periodically tested by NIST; and

A user who documents derived materials with stated uncertainty and whose value has been verified with analytical and measurement systems that have been periodically tested through an unbroken chain of comparisons to the national physical standards.

Periodic testing of source manufacturers, whether they be commercial or agency suppliers or end users, is most cost effectively implemented through measurement assurance programs that are ultimately linked to NIST traceability (Hoppes, 1990).

A comprehensive discussion of germanium detector set up and calibration can be found in ANSI N42.14.

15.10 Other Considerations

15.10.1 Alpha

15.10.1.1 Troubleshooting

A number of factors can influence alpha counting results. These include attenuation or self absorption, detector contamination, and other radionuclide interference. Attenuation or self absorption corrections need not be made if constant conditions are maintained for sample and calibration standard counting. If conditions can not be held constant, then corrections will have to be made in order to produce accurate results. For example, the gamma rays from $^{137}$Cs in a water matrix counted in a 90 cm$^3$ aluminum can will require a 15 percent correction. Individual electrical line conditioners or uninterruptible power supplies as well as supplemental air conditioning can be provided in the counting rooms to maintain electrical and environmental stability. Additionally, humidity control can also be provided. Temperature and humidity may be recorded with a chart recorder.

Detector contamination can also be a problem in some cases and, therefore, detector backgrounds should be periodically checked. Contaminated detectors will have higher background counts and even when sample spectra are corrected for the presence of contamination the higher background results in higher MDAs. Finally, some alpha counters may be sensitive to beta radiation, and corrections may have to be made for this interference. For a routine operating alpha counting
system periodic instrument QC checks should be performed at some specified frequency. This would include, as appropriate, counting efficiency, background, resolution, gain, and voltage plateau.

Solid state detectors used for alpha spectrometry can become contaminated by recoil. This recoil contamination, which increases the detector background, takes place when fragments from sources travel to the detector and are implanted in the detector surface by the recoil energy imparted to the nucleus of an alpha-emitting atom. The energy of the fragments may be sufficient to implant them in the detector so that they cannot be removed non-destructively. Recoil contamination can invalidate a count after only a single sample count and cause a constant need to decontaminate equipment.

The application of a negative bias to the sample, in conjunction with an absorbing layer of air, or a thin film absorber (12 µg/cm³) helps to keep recoil particles from imbedding themselves into the detector. For better resolution and where recoil contamination is of no concern, it is advisable to maintain a low pressure. Typically, systems can pump down to under 50 µm and, by continuously running the pump, maintain that level indefinitely.

Detector contamination dominated by two processes, alpha recoil and “volatilization” of polonium. Alpha recoil contamination occurs when an alpha-emitting nuclide on the source plate decays to an alpha-emitting daughter or string of progeny. Since the specific activity is inversely proportional to the half-life for a fixed number of atoms, recoil will produce the most background activity when relatively short-lived progeny are produced. However, if the half-lives in question are very short (say up to a few hours), they will decay away quickly enough to be of little concern in alpha spectrometry. Particularly serious are those cases that involve transfer of recoil progeny with half-lives from days to weeks, short enough that a reasonable amount of parent activity will produce a significant amount of recoil contamination, and long enough that decay back to normal background levels will require an inappropriately long time. In addition, the effect is chronic: similar recoil-producing samples counted in the same chamber will produce a long-term build-up of detector background which could eventually become serious.

Some common examples of decay-chains that produce recoil contamination include $^{228}\text{Th}$, $^{229}\text{Th}$, and $^{226}\text{Ra}$. It is important to realize that even β-emitting nuclides ejected by alpha recoil can contribute to alpha background if they subsequently decay to alpha emitters. For example, the direct daughter of $^{229}\text{Th}$ is $^{225}\text{Ra}$ which decays by β-emission to the α-producing daughter $^{225}\text{Ac}$.

Contamination of detectors by polonium isotopes, such as $^{210}\text{Po}$ (τ₀ = 138.4 days), should occur by some other process than alpha recoil. Note that $^{210}\text{Po}$, the last radioactive member of the $^{238}\text{U}$...
decay series, is the daughter of $^{210}\text{Bi}$, a beta-emitter. The transfer of polonium from a source to a silicon detector has been attributed to “aggregate” recoil and inherent volatilization of polonium at low pressure. Whatever the actual cause, it is clear that polonium activity is indeed transferred to detectors, a very serious problem with long-lived $^{210}\text{Po}$ and even worse when working with $^{209}\text{Po}$ ($t_{1/2} = 102$ years) as a yield tracer.

Liquid Scintillation Quenching

Quenching, which is probably the most prevalent interference in liquid scintillation counting, can be defined as anything which interferes with the conversion of radionuclide decay energy to photons emitted from the sample vial, resulting in a reduction of counting efficiency. Two types of quenching may be encountered in liquid scintillation counting: optical or color quenching and chemical quenching. Color quenching results in a reduction in the scintillation intensity (as seen by the multiplier phototubes) due to absorption of the scintillation light by materials present in the scintillation solution resulting in fewer photons per quanta of particle energy and a reduction in counting efficiency. Chemical quenching results in a reduction in the scintillation intensity due to the presence of materials in the scintillation solution that interfere with the process leading to the production of light resulting in fewer photons per quanta of particle energy and a reduction in counting efficiency. The quenching process may be illustrated as follows:

$$\text{Radionuclide Decay} \rightarrow \text{Beta} \rightarrow \text{Solvent & Scintillator} \rightarrow \text{Light Photon} \rightarrow \text{Multiplier Phototube}$$

| Chemical Quench | Chemical Quench |

One can have both types of quenching present in a sample. Note that in chemical quenching all energy radiations are equally effected, but in color quenching not all energy radiations are equally effected. Therefore, the measured sample counts should be corrected for quenching effects so that the radioactivity in the sample can be quantified. Typical quench corrections include Channels Ratio, External Standard and Internal Standardization.

Attenuation

Attenuation or self absorption corrections may be necessary for alpha counting. Attenuation corrections should be made whenever the sample matrix differs from that of the calibration standard. For example, when a gross alpha analysis is performed on an evaporated water sample of some thickness and an electroplated standard was used for the calibration. Attenuation corrections will have to be made. Alpha particle attenuation corrections will generally be necessary with a sample density thickness greater than about one mg/cm$^2$. 

Figure 15.10 shows how severe the attenuation of alpha particles is in air.

15.10.1.2 Calibration

Alpha counting instrumentation should be calibrated with the specific radionuclide of interest or a radionuclide of similar alpha energy under the same configuration that the sample will be counted. The standard should contain the same solid material as the sample and be of the same weight. If the samples and calibration standard are not counted under identical conditions, then corrections will have to be made. Also, if there is a variation in weight from sample to sample corrections will have to be made, typically a calibration curve relating sample weight to counting efficiency is used.

Alpha calibration standards are available from NIST or NIST-traceable commercial vendors. Among the radionuclides available are $^{230}$Th, $^{241}$Am, $^{235}$U, $^{239}$Pu, $^{228}$Th, $^{238}$U, and $^{226}$Ra. Other radionuclides are also available, NIST or a commercial vendor should be contacted regarding procurement. Sources should be prepared in the manner in which the sample will be counted. The source may be procured as a solution and then prepared in the appropriate counting geometry, or the source may be procured directly in the appropriate geometry, such as an electroplated standard.

15.10.1.3 Costs

There are three major types of detectors used for alpha counting. Their cost will depend on the type of information wanted and the number of detectors in the unit.

Solid state silicon surface barrier detectors are used to count and distinguish alpha particles of different energies. An alpha spectrometer consists of a vacuum chamber, detector, electronics to amplify the signal, a multichannel analyzer, and some means of collecting data. A system with
eight detectors, vacuum pump, computer and the software necessary for data collection and data reduction costs approximately $50,000.

A liquid scintillation counter can be used to count alpha particles and in some cases provide some information about the energy distribution, although with poorer resolution than silicon surface barrier detectors. The LSC unit is typically set up to count samples sequentially, using one detector and automatic sample changer. The price depends on the background required, and will range from $25,000 to as much as $45,000. This price includes a computer and the appropriate software.

A gas-flow proportional counter is used to count samples for a gross alpha (or beta) activity. The price of a unit depends on the number of detectors, the size of each detector, and the accessories. One major accessory could be an automatic sample changer. A system with 8 to 10 small detectors (1 inch in diameter) will cost from $35,000 to more than $60,000.

There are no maintenance costs associated with an alpha spectrometer. If properly used and monitored, the system will retain its specifications for a long time. The detectors may need replacing eventually, if its resolution deteriorates or it becomes contaminated, at a cost of $500-$1,000 each.

A liquid scintillation counter requires the use of an organic scintillation cocktail, which cannot be reused. The total cost of this cocktail, combined with the cost of the sample vials, should not exceed $500 for an annual throughput of approximately 1,000 samples.

The operation expense associated with the use of a gas-flow proportional counter is for the ultra-high purity P10 gas, which is necessary if stable efficiencies and low backgrounds are required. All proportional counters should have calibrated gas regulators for accurate and reproducible settings of flow rates. The flow rate should be placed with the QC information that is with the other instrument QC. For almost constant operation of a system with eight detectors, as many as 24 tanks of P10 gas per year will be required, at a total cost of approximately $7,000.

All of the above instruments should be in a fairly constant temperature and low humidity environment, so that air conditioning and/or heating costs need to be factored in, as needed.
15.10.1.4 Quality Control

Statistical quality control (SQC) is discussed here to familiarize the reader with its application to nuclear counting instrumentation. More detailed information about SQC is provided in Chapter 19.

The primary tool for statistical quality control is the control chart. A control chart is a graphical tool for monitoring the distribution of values produced by a measurement process or system. The distribution of values observed during a period when the system is in statistical control is used to set up the control chart. Subsequent values are then plotted on the chart and inspected to ensure that the system remains in control.

Typically one or more control charts for counting efficiency and background are maintained for each counting instrument. The instrument should be fully operational before the control charts are implemented. However, control charts should be in use before calibration of the instrument for a particular analysis to ensure that the instrument parameters are in statistical control during the calibration.

The selection of the check source for monitoring counting efficiency is critical and should be made after considering guidance in this document. The source geometry, half-life, and radiation energy are important factors.

A control chart should be based on an initial data set obtained from at least 15 measurements. Ideally, at least 10,000 counts per measurement are recommended to provide a relative counting uncertainty of no more than 1 percent. For some instruments, achieving the recommended 10,000 counts may be impractical, especially for a background control chart. It may also be undesirable to place a high-activity efficiency check source in a low-background detector because of the potential for contamination.

The initial measurements should represent the measurement system as it is used over time. Making the measurements over several days ensures that variability due to temperature and humidity changes is included. The source should be repositioned before each measurement to ensure that variability due to positioning error is included.

The mean and standard deviation of the counts or count rates are estimated from the initial data set. The mean is used as the central line (CL) of the control chart. Warning limits are placed at ±2 standard deviations from the central line, and control limits are placed at ±3 standard deviations from the central line.
Statistical tests of the data distribution should be performed at the time the warning and control limits are established. Tests for normality are common. It is also common to test whether the counts follow the Poisson model (Chapter 19).

The central line and warning and control limits for an efficiency control chart should be adjusted for radioactive decay of the check source unless the source is very long-lived. Either the limits or the data points may be decay-corrected. It may also be necessary to adjust the counting time for the check source measurements if the source decays considerably during the period when the chart is in use. It is important to note that the relative standard deviation of the measured values increases as the mean number of counts per measurement decreases.

When a measured value falls within the warning limits, the measurement system is considered to be in control. If a value falls outside the control limits, the system is considered out of control. These two rules are commonly used to evaluate control charts, although stricter evaluation criteria are sometimes used. Common sense should be exercised if the data meet the objective evaluation criteria but nevertheless demonstrate patterns or trends that might indicate developing problems. For example if a long increasing or decreasing sequence of values is observed, an investigation is probably warranted even if all of the values are between the warning limits.

Generally, if a value falls within the control limits but outside the warning limits, the system may require more attention but it is not yet considered definitely out of control. The Westgard Rules, which are recommended by ASTM E1329, provide more elaborate criteria for evaluating such measurements.

### The Westgard Rules*

1. Is the measurement more than 2 sigma from the mean? If not, go to Step 7.
2. Is the measurement more than 3 sigma from the mean? If so, go to Step 8.
3. Are the last two measurements more than 2 sigma from the mean? If so, go to Step 8.
4. Is the range of the last two measurements more than 4 sigma? If so, go to Step 8.
5. Are the last four measurements more than 1 sigma from the mean? If so, go to Step 8.
6. Are the last ten measurements more on the same side of the mean? If so, go to Step 8. Otherwise, go to Step 7.
7. Accept the measurements. Stop.
8. The measurements are out of control. Stop.

*Adapted from ASTM E1329.

The following two sections on proportional counting and liquid scintillation counting are applicable to both alpha and beta measurements.
Proportional Counters

The following should be considered when QC checks are not within limits.

1. Is the standard decay corrected, correctly?

2. Check log book to see what changes were made to counter and if the repairman recently changed any switch settings.

3. If gas cylinder was changed recently, was system allowed to purge? Was correct gas (¹⁰⁵P) obtained? Verify the correct regulator pressure, and ensure the gas cylinder valve is open all the way.

4. If backgrounds are high, check for dirt or dust on the background planchet. Check window for contamination and replace if necessary.

5. Check alpha and beta voltages.

6. Check discriminator settings.

7. Check voltages on nim bin power supply (±12V, ±24V).

8. Check alpha and beta plateau voltage for drift.

Liquid Scintillation Counters

The following should be considered when QC checks are not within limits.

1. Is the standard decay corrected, correctly?

2. Has the quench value for the unquenched standard for the instrument changed? The quench value for the unquenched standard indicates the overall gain of the system. Run the autocalibration and verify the result with the historical result.

3. Check for dirt or fingerprints on outside of vial.

4. Check for dirt inside instrument.
5. Is sample two phase?

6. Has standard dark adapted and reached temperature equilibrium?

7. Check log book to see what changes were made to machine and if repairman recently changed any switch settings.

15.10.2 Beta

15.10.2.1 Introduction

Accurate beta particle measurements will depend upon the degree and extent to which the parameters that affect the measurement process under considerations are quantified. These parameters may include:

- Radiation detector used;
- Material and shape of the final sample mount;
- Form and thickness of final sample for analysis;
- Radionuclide purity of final sample;
- Final sample-to-detector distance; and
- Beta particle energy.

Beta particle attenuation or self absorption corrections to the detector efficiency may be necessary depending on the beta particle energy detection system and final sample form. The potential of detector contamination from sample measurements is a function of the type of detector used and the stability of the final sample composition. The inherent beta particle background of the various detection systems should be evaluated and its contribution removed from the sample measurement result. The beta particle measurement system should be calibrated with NIST-traceable standards and its subsequent performance held to established measurement quality requirements through the use of daily or prior-to-use quality control checks. In addition, appropriate instrument quality control should be established for background, voltage plateau, quenching, resolution and alpha-beta cross talk. Guidance on beta particle counting can be found in industry standards (ASTM D1890; D3648; E1329) and publications (NCRP Report 58; Knoll, 1989; Lapp and Andrews, 1954; Price, 1989; USPHS, 1967).

“Gross” alpha and beta counting of evaporated samples, wherein a multitude of alpha and beta-emitting radionuclides may exist, is typically used for screening of water samples. The application of such methods may be targeted for a specific radionuclide or a category of
radionuclides such as the naturally occurring nuclides or a specific radionuclide in a facility
effluent. However, extreme caution should be applied to the interpretation and use of such results
without a full specific radionuclide characterization of the water source under investigation. The
type of analysis is to be considered “gross” and, in most cases and for a variety of sound
technical reasons, the gross measurement result does not equal the sum of the radionuclides
contained in the sample.

When specific radiochemistry is performed the beta-emitting radionuclide of interest will be
isolated, concentrated and converted to a desired final chemical and physical form. Under these
circumstances, the beta detection system should be calibrated for the radionuclide, chemical
composition of the final sample form and the range of final sample weights expected from
chemical recovery.

15.10.2.2 Alpha Particle Interference and Beta Energy Resolution

When properly operated or under optimal counting conditions (thin final samples or low LS
quenching and high beta energy), most beta particle counting systems can separate alpha and beta
particle detection events. However, the degree of alpha particle detection by the beta detector
under consideration should be evaluated for each radionuclide, mixture of radionuclides or
specific final sample form. Beta detection systems that are considered to have beta energy
spectral resolution capabilities may be less affected by samples containing alpha-particle emitting
radionuclides. However, for window gas proportional counters, alpha particle energy degradation
by air, detector window or sample self absorption may lead to false beta detection without proper
evaluation. A typical example would be a thick final sample matrix containing a mixture of alpha
and low-energy beta-emitting nuclides.

Some commercial window gas proportional counting systems have a feature for simultaneous
alpha and beta particle counting that uses a voltage pulse height discrimination for the separation
of beta and alpha particle detection events. A common and more historical means of separating
alpha and beta particle events is to count the sample on the alpha proportional counting voltage
plateau followed by a count on the beta (plus alpha) proportional counting voltage plateau. An
alpha-to-beta crosstalk factor should be determine for the final sample weight and for the alpha
and beta energies under consideration. The net beta count is determined by multiplying the alpha
counts (from the alpha window for simultaneous counting or on the alpha counting plateau) by
the alpha-to-beta cross talk factor.

Window gas proportional counters typically are not used for beta spectrometers but instead
record beta particle detection events giving rise a voltage pulse large than a discriminator setting.
Under most circumstances, liquid scintillation counters have sufficient energy resolution capability and electronic discrimination to fully separate beta and alpha particle detection events. However, due to the nature of the beta energy continuum of an emission process and the inherent resolution of a liquid scintillation spectrometer, identification and quantification of multiple nuclides contained in the same sample is complicated unless their beta energies are widely separated. Computer software and beta interference factors should be applied in such cases.

A liquid scintillation counter is typically used for Cerenkov counting. However, the final sample solution contains no scintillator as would a full liquid scintillation-sample cocktail. Cerenkov counting, due to the nature of measurement process, will not detect alpha particles of any energy or beta particles having an average beta energy less than 260 keV. Cerenkov counting is typically applied to single nuclide evaluations or for a mixture of two nuclides that have a differential maximum beta energies greater than 700 keV (e.g., $^{89}$Sr and $^{90}$Y). Beta interference factors should be applied in such cases.

15.10.2.3 Liquid Scintillation Quenching

The information on liquid scintillation quenching provided in Section 15.10.1.1 is applicable for beta particle detection. The degree of quenching should be determined for each radiochemical method, radionuclide or application. An appropriate correction factor/curve should be calculated and applied to the measurement results for the samples being evaluated. The magnitude of the quench correction may approach 50 percent in certain severe quenching situations.

Cerenkov counting is less sensitive to “quenching” than liquid scintillation counters using scintillation cocktails. Typically, the final sample solution is a result of a control radiochemical process that eliminates most sources of contamination, chemical impurities and variability in the final sample solution.

15.10.2.4 Beta Particle Attenuation

Beta particle attenuation should be considered for window gas proportional, plastic scintillator and solid state detector counting applications. Beta particle attenuation can result from the interaction of a beta particle with the air, detector window or the matrix atoms of the final sample. Beta particle air attenuation is a function of the distance between the sample or source and the detector’s particle entrance window. Under most application for beta particle counting, this factor is typically insignificant compared to the other sources of beta particle attenuation.
Figure 15.11 shows the attenuation of beta particles is in air and water. Consideration of the detector window thickness and its beta particle attenuation becomes important when evaluating low energy beta particles such as $^{14}$C. Normally, the air and detector window attenuation factors are determined as a combined beta attenuation-efficiency factor that includes the sample self absorption for a given application. In most applications, a back scatter factor for the material composition (Z value) of the final sample mount is included into a combined attenuation-backscatter-efficiency factor or, more simply, the combined detector efficiency correction factor.

For the lower to intermediate beta particle energies, the combined detector efficiency factor is a function of beta energy, final sample mass and mass composition. For beta particles having a maximum beta energies greater than 1,500 keV, the combined detector efficiency factor is nearly constant over a final sample weight range of 0 to 5 mg/cm². A typical combined beta detector efficiency curve for $^{131}$I (606 keV $\beta_{\text{max}}$) as CuI over a weight range of 0 to 50 mg is shown for a plastic scintillator beta detector in Figure 15.12. A complete review of the detection method can be found in reference (McCurdy et al., 1980).
15.10.2.5 Calibration

Beta particle counting systems should be calibrated with the specific radionuclide under investigation or a surrogate radionuclide of similar beta energy having a comparable final sample composition and configuration. However, it should be mentioned that moderate to severe calibration biases may occur depending on the severity of the departure from the chemical composition of the final sample matrix and the beta energy of a surrogate. For this reason, the use of a surrogate radionuclide is discouraged unless the availability of the radionuclide of interest is non-existent. Corrections between the surrogate and radionuclide of interest should be determined and applied to sample results. For electroplated plated samples, a correction factor needs to be determined if the plating material of the surrogate is not the same as that used for the samples.

Cerenkov counting normally involves a single radionuclide calibration (single energy calibration) for the final sample solution. Typically, the final sample solution is a result of a control radiochemical process that eliminates most sources of variability for the calibration process.

Aqueous beta-emitting radionuclide calibration standards and sources are available from NIST or from a NIST-traceable commercial radioactive source manufacturers. The long-lived pure beta-emitting radionuclides available from NIST include: \(^3\text{H}, 14\text{C}, 63\text{Ni}, 129\text{I}, 89\text{Sr}, 90\text{Sr}, 99\text{Tc}, 228\text{Ra},\) and \(^{241}\text{Pu}\). The majority of the gamma-emitting radionuclides also emit beta particles in the nuclear transformation process. Check Section 15.4 for the availability of known beta- gamma emitting radionuclides. Contact a NIST-traceable radioactive source manufacturer for the availability of other pure beta or beta/gamma-emitting radionuclides (ANSI N42.15, American National Standard Check Sources for and Verification of Liquid-Scintillation Counting Systems).

Aqueous radioactive standards can be prepared in the appropriate geometry for LS or Cerenkov counting or through chemical processing precipitated or electroplated as final sample form for counting by a gas proportional, plastic or solid state beta detection system.

15.10.2.6 Costs

There are four principal beta detection methodologies available. Window gas proportional counting and liquid scintillation counting systems (Cerenkov counting as well) can be purchased with the option of readily available automatic sequential sample counting systems. Sample capacity is typically 100. These automatic sequential counting systems are available in the $30,000 to $50,000 range depending on options. Multiple detector window gas proportional counters having a simultaneous counting capability are available from some commercial
manufacturers. The basic unit contains four detectors but several units can be combined to give eight or 16 detector systems. The basic price for such units is in the range of $20,000 to $50,000 depending on the number of detectors and options.

Solid state silicon surface barrier and ion implanted Ge or Si detectors are used to perform spectral analysis of beta emitting radionuclides. A solid state beta spectrometry system consists of a vacuum chamber, solid state detector, high voltage-preamp-amplifier instrumentation modules, a multichannel analyzer (MCA) or equivalent computerized MCA using an analog-to-digital converter and electronic data storage. Individual ion-implanted Ge detectors having an active area of 450-2,000 mm² and a 500 µm thickness range in price between $1,300 and $3,200. Beta resolution of these detectors is typically approximately 12 keV.

A beta spectrometry system consisting of eight detectors with vacuum pump and computer would be approximately $30,000-$40,000, without background reducing shielding. Solid state spectrometry systems for beta particle applications, unlike that for alpha particles, would be sensitive to external background from cosmic radiation, terrestrial radiation and inherent beta radioactivity in the surrounding materials.

Automatic sample counting, plastic scintillator beta particle detection systems have not been commercialized for the radioassay laboratory setting. Most of these systems have been fabricated by the user from readily available components, electronic modules, multichannel analyzers and lead shielding. The cost of a single detector system is estimated to be less than $15,000.

Maintenance costs for the liquid scintillation counters, window gas proportional counters and alpha spectrometry systems have been discussed in Section 15.10.1.3.2 for alpha counting applications. If a laboratory already has existing units for the alpha particle measurement applications, there will be no additional maintenance cost relative to their use for beta particle measurements.

There is no maintenance cost associated with the operation of a plastic scintillator beta spectrometry system.

Costs associated with the maintenance of the room environment for the nuclear detection equipment should be considered. Service maintenance relative to the constant voltage supply or uninterruptable power sources as well as having a dust free constant temperature and humidity environment should be considered.
15.10.2.7 Quality Control

See section 15.10.1.4.

15.10.3 Gamma

15.10.3.1 Troubleshooting

Once a gamma-ray spectrometry system has been established in accordance with the manufacturer’s or supplier’s instructions, a daily count of a calibration or reference source should be performed to assure the system continues to operate properly. The three parameters that should be checked and recorded are: energy calibration (keV/channel), counting efficiency (count rate/decay rate), and gamma-ray peak resolution (FWHM). With the exception of a complete detector or electronic component failure (no pulses are detected at the preamp or multiplier phototube output), degradation of gamma-ray peak resolution will be the first indication that detector is not performing properly or that electronic noise has been introduced into the counting system by the preamplifier, amplifier, or multichannel analyzer. Any indications that the detector efficiency is not within statistical limits of expected values should be reported, since this value will be used to convert the observed count rate to decay rate. The energy calibration should either be recorded with sample spectral data or adjusted daily to a previously established constant value. This energy calibration should be accurately known so that nuclide identifications can be made. See page 51 for a list items to be checked if the counting system is out of specifications.

Gamma-ray spectrometry systems are extremely sensitive to both electronic and environmental conditions. Temperature changes can cause spectral shifts and improper nuclide identifications because of incorrect energy calibrations. Excessive humidity in the detector preamplifier can cause high voltage arcing which results in poor peak resolution or complete system failure. Improper pole zero settings, which affects the shape of the pulses being analyzed, can cause degradation of peak shapes and resolution. Poorly conditioned NIM power can introduce electronic noise which will also result in degraded peak resolution. Routing of cables between the detector, electronics, multichannel analyzer, computers, and monitors is very important. The introduction of any spurious electronic noise into any of the components that make up the gamma-ray spectrometry system can degrade the resulting data.

The need to make corrections for self-absorption in environmental samples during routine gamma-ray spectrometry cannot be overemphasized (Modupe et al., 1993). The correction to be made for the difference in self-absorption between calibration standards and sample matrices is usually small for intermediate and high energy photons, but it is not negligible at low energies.
where the photoelectric effect is the most important mode of attenuation. The photoelectric process varies approximately as $Z^{4.5}$ ($Z$ is the atomic number of the elements in the medium) so that a change in the elemental composition of a sample relative to a calibration standard can require a correction factor for detector efficiency as high as a factor of 2.

\[ \frac{I}{I_o} = \frac{1 - e^{-\mu H}}{\mu H} \]  \hspace{1cm} (15.1)

The quantities $\mu$ and $H$ are the linear attenuation coefficient and the thickness of sample, respectively. $I_o$ and $I$ are the intensities of the beam emerging from the sample container without and with an absorbing matrix in place. This is the traditional self-absorption equation. For complex counting geometries of homogeneous materials, an estimated average $H$ (sample thickness) can be used.

The method for self-absorption correction at various energies requires that the linear attenuation coefficient, $\mu$, of the sample matrix be known. Knowledge of $\mu$ usually requires that the elemental composition of the matrix be determined. The tedium and time required in elemental analysis may make it impractical for routine gamma-ray analyses involving large numbers of samples. Computer programs are available to calculate $\mu/\rho$ for various compounds when the percent elemental composition of the compound is known. $\mu/\rho$ is computed as a linear combination of the mass attenuation coefficients of the composite elements.

\[ \mu/\rho = \sum (\mu_i/\rho_i) P_i \]  \hspace{1cm} (15.2)

where $P_i$ is the percent by weight of the $i$th element in the compound.

The gamma-ray path length, $H$, is equal to the thickness of sample. When performing gamma-ray transmission measurements to determine $\mu$ a path length of $H$ is used. To determine the self-absorption correction for radioactive samples, the corrections are integrated for a path length of 0 to $H$.

When a photon beam passes through a homogenous sample of mass attenuation coefficient, $\mu/\rho$, density, $\rho$, and thickness, $H$, the percentage beam attenuation, $A$, is given by

\[ A = \frac{I_o - I}{I_o} \times 100\% = (1 - e^{-(\mu/\rho)H\rho}) \times 100\% \]  \hspace{1cm} (15.3)
15.10.3.2 Calibration

Most gamma-ray spectrometry systems are calibrated with either single or mixed standards in an exact matrix and geometric form as the samples to be analyzed. However, there are computer codes that can calculate detector efficiency from the physical dimensions of the detector and sample counting geometry (Mitchell, 1986 and 1988, Hensley et al., 1997). Commercial standards of single or mixed gamma-ray emitters in a matrix of known chemical composition and density can be prepared in user supplied containers. Calibrations based upon these standards can then be adjusted to correct for any differences in composition and density between the calibration source and the sample (Modupe et al., 1993).

Table 15.2 lists some gamma-ray emitting nuclides that can be used for energy and efficiency calibration (Sanderson et al., 1993; Browne et al., 1986).

<table>
<thead>
<tr>
<th>NUCLIDE</th>
<th>ENERGY (KeV)</th>
<th>HALF-LIFE</th>
</tr>
</thead>
<tbody>
<tr>
<td>$^{210}$Pb</td>
<td>46.5</td>
<td>22.3 years</td>
</tr>
<tr>
<td>$^{241}$Am</td>
<td>59.5</td>
<td>432.2 years</td>
</tr>
<tr>
<td>$^{109}$Cd</td>
<td>88.0</td>
<td>462.6 days</td>
</tr>
<tr>
<td>$^{57}$Co</td>
<td>122.1</td>
<td>273 days</td>
</tr>
<tr>
<td>$^{141}$Ce</td>
<td>145.4</td>
<td>32.5 days</td>
</tr>
<tr>
<td>$^{139}$Ce</td>
<td>165.9</td>
<td>137.7 days</td>
</tr>
<tr>
<td>$^{203}$Hg</td>
<td>279.2</td>
<td>46.6 days</td>
</tr>
<tr>
<td>$^{51}$Cr</td>
<td>320.1</td>
<td>27.7 days</td>
</tr>
<tr>
<td>$^{113}$Sn</td>
<td>391.7</td>
<td>115.1 days</td>
</tr>
<tr>
<td>$^{85}$Sr</td>
<td>514.0</td>
<td>64.8 days</td>
</tr>
<tr>
<td>$^{137}$Cs</td>
<td>661.7</td>
<td>30.0 years</td>
</tr>
<tr>
<td>$^{54}$Mn</td>
<td>834.8</td>
<td>312.5 days</td>
</tr>
<tr>
<td>$^{88}$Y</td>
<td>898.1, 1836.1</td>
<td>106.6 days</td>
</tr>
<tr>
<td>$^{65}$Zn</td>
<td>1115.5</td>
<td>243.8 days</td>
</tr>
<tr>
<td>$^{60}$Co</td>
<td>1173.2, 1332.5</td>
<td>5.27 years</td>
</tr>
<tr>
<td>$^{40}$K</td>
<td>1460.8</td>
<td>1.28 x 10^9 years</td>
</tr>
</tbody>
</table>
15.10.3.3 Software

Most laboratories are now using commercially available software for the analysis of gamma-ray spectra. These programs are easy to use and do not require the user to be an expert in gamma-ray spectrometry. An evaluation of some of these programs in 1987 indicated there were substantial differences in the abilities of the programs to resolve multiplets of unequal intensity and to analyze complex spectra (Sanderson 1988). Another evaluation was completed in 1992 (Decker and Sanderson, 1992) since many of the programs had undergone numerous revisions and there were a few new programs available. The second evaluation indicated a substantial improvement in the deconvolution of doublets and the results of the analysis of a Chernobyl air filter were much more consistent than when a similar filter was analyzed in the first evaluation. The six programs analyzed in 1991 include GAMMA-W from Germany, INTERGAMMA from France, OSQ/Plus from Canada, SAMP090 from Finland (supplied by Canberra Industries, USA) and OMNIGAM and GDR from the United States. Some of the features which contribute to a good program included the ability to display the spectrum as well as calculated calibration files, the ability to manually insert peaks during the fitting procedure, an extensive nuclide library and the ability to easily transfer nuclides to smaller, working libraries, an analysis report which includes the names of the calibration files used, a peak fit report including any problems with the shape of the peaks, and identification of the peaks used in the activity calculation as well as any problems with interfering lines.

In 1996 the Environmental Measurements Laboratory of the U. S. Department of Energy began a Gamma Spectra Data Evaluation program (Decker et al., 1996) whose goal was to test the ability of the present day software to accurately identify and quantify the nuclides in a complex spectra and the ability of the user to properly utilize the software. In order to do this, synthetic spectra were generated using the computer code SYNTH developed by Walt Hensley at the Pacific Northwest National Laboratory. The spectra were then converted to a variety of formats on disk and Digital Equipment Corporation (DEC) TK 50 tape and sent to DOE laboratories and DOE contractors. A calibration spectrum, a background spectrum and three sample spectra were sent to each participant. These spectra simulated those that would be obtained when an air filter was counted 10 cm from a 22 percent coaxial detector with a 0.5 mm beryllium window. Two of the samples contained fallout and naturally occurring nuclides with half lives greater than thirty days. The third sample contained both short and long lived fission product nuclides. Thirty one laboratories participated using 16 different software packages. The software packages included Aptec, Vertechs GDR/P, Nuclear Data ASAP, various Ortec packages, and various Canberra packages for both the PC and the DEC MicroVax. Most of the laboratories did fairly well with the first two samples. A few laboratories reported nuclides that were not present in the third sample and did not accurately quantify those that were. The results did not seem to be software...
dependent but were due to the user utilizing or not utilizing available software features properly. There was quite a wide range of numbers for both the uncertainty terms and for the minimum detectable activities which seems to indicate we need a consistent way of calculating these terms to make them more meaningful.

15.10.3.4 Costs

Gamma-ray spectrometry systems can cost from $14,000 to well over $60,000 depending upon the choice of detector. For a 75 x 75 mm NaI(Tl) system the costs would be approximately $1,000 for the detector, $5,000 for a 10 cm graded lead shield, and $8,000 to $10,000 for a multichannel analyzer. Very large HPGe detectors will cost more than $50,000. The actual detector cost will depending upon the size of the germanium crystal, its resolution, and method of cooling. Data reduction costs (software and computer) would be an additional expense for either type of system.

NaI(Tl) detector systems do not require any additional maintenance beyond what any laboratory electronic system requires. Each HPGe detector will require approximately $1,200 per year for liquid nitrogen to maintain their operating temperature. An electrical/mechanical cooler can be used in place of a liquid nitrogen cryostat but it will require 0.5 to 1.0 kW of power around the clock to operate. Both systems should be operated at constant temperature for reliable performance. This may require substantial air conditioning.

15.10.3.5 Quality Control

Initial data to prepare solid state gamma detector QC charts may be obtained by counting a mixed gamma point source between 20 to 30 times (Ideally, these counts should be over a period of several weeks. However, if time does not permit, the counts may be accumulated over 1 to 7 days.) Two or three QC charts (depending on age of mixed gamma point source) are initially established for the mixed gamma point source and control limits are established for background. The three source charts cover the low energy (88 keV, $^{109}$Cd), the medium energy (661.6 keV, $^{137}$Cs), and the high energy (1,332.5 keV, $^{60}$Co). The source is counted until between 10,000 to 40,000 counts are obtained in each photopeak.

Background QC charts are established according to the procedure already listed for Proportional and liquid scintillation counters with the following exception: the background is counted and the total counts in the spectrum are obtained by summing the counts in the entire spectrum.
The resolution of the detector (FWHM) is measured each month and recorded, but it is not plotted. A NIST $^{60}$Co source is positioned 25 centimeters from the end-cap face and counted for 100 minutes. The FWHM is calculated by the peak search program for the 1,173.2 keV and 1,332.5 keV peaks, and recorded in the logbook.

When the energy of the source QC exceeds the specified energy tolerance (for example ±0.75 keV) from its initial calibrated value, the analyzer system should be recalibrated. First determine whether a gain or zero shift has occurred. A gain shift is a nonlinear shift in channels of low and high energy peaks (i.e., $^{109}$Cd peak shifts ±1 channel and $^{60}$Co peak shifts ±3 channels). A zero shift is a linear shift in channels for both low and high energy peaks (i.e., $^{109}$Cd peak shifts ±1 channel and $^{60}$Co shifts ±1 channel also). Make the appropriate adjustments to the amplifier (gain) or the Analog to Digital Converter (zero). Recalibrate the analyzer and record the slope (keV/channel) and the zero intercept in the log book. If the best fit of the recalibration curve is a nonlinear fit (quadratic), record the “Q” coefficient, keV/channel$^2$, in the log book. Also record the updated FWHM calibration factors, slope, offset, and FWHM at 1,332.5 keV in the log book.

The following should be considered when QC checks are not within limits.

- Is standard decay corrected to the proper date?
- Check sample positioning.
- Check for zero shift.
- Check for gain shift.
- Check full width at half-maximum.
- Check nim bin power supply voltages (±6 V, ±12 V, ±24 V).
- Check efficiency tables.
- Check for moisture on the detector due to recently filling the dewar with liquid N$_2$.

**15.10.4 Non-Nuclear Instrumentation**

**15.10.4.1 ICP-Mass Spectrometry**

ICP-MS is one of the most versatile and sensitive atomic spectroscopy techniques available. It can be used to determine the concentrations of over 70 elements. The detection limit of the technique extends down to the parts-per-billion range in soils and to the parts-per-trillion range in waters. This sensitivity makes ICP-MS an attractive complement to decay-counting techniques in the radiochemical analysis laboratory. For very long-lived radioisotopes (those with half-lives over 10,000 years, e.g., $^{244}$Pu, $^{99}$Tc, $^{129}$I), ICP-MS may be faster and more sensitive than decay counting. In addition, sample preparation for ICP-MS can avoid some of the analyte separation
and purification steps required for decay counting, providing an additional dimension of time
savings. Another important feature of ICP-MS is its ability to provide isotopic distribution
information (e.g., $^{238}\text{U}$ vs. $^{235}\text{U}$). This information is frequently useful in determining the age
and/or origin of materials. (ASTM C758, C759, C799)

The isotopic discrimination capabilities of ICP-MS make possible the calibration technique
known as isotope dilution. In this procedure, a sample is analyzed for one isotope after having
been spiked with a different isotope of the same element (e.g., analysis of $^{235}\text{U}$ might involve
spiking with $^{233}\text{U}$). The spiked sample is carried through all preparation and analysis steps; in this
way, any matrix or procedural effects that might influence the $^{235}\text{U}$ signal will influence the $^{234}\text{U}$
signal to precisely the same extent. Final quantization relies on measuring the ratio of unknown
(here the $^{235}\text{U}$ signal) to the known ($^{234}\text{U}$) signal. Isotope dilution is a way of generating highly
precise and accurate data from a mass spectrometer and has been used in the characterization of
many certified reference materials.

Although an ICP-MS instrument is extremely delicate, with proper care and preventive
maintenance system up time should range between 80 to 95 percent. An initial investment of
about $200,000 will be required to obtain a current commercial state-of-the-art system. Annual
maintenance costs will run from $5,000 to $20,000 depending on the purchase of a service
contract.

For more sophisticated measurements, at substantially higher cost, an ICP-MS with magnetic
sector, instead of quadrupole, detection can be applied. Sector instruments are capable of
resolving species of very similar mass. For example, $^{99}\text{Tc}$ might be resolved from a
contamination of $^{99}\text{Ru}$ with a high-resolution mass spectrometric detector. More typically, high
resolution instruments are employed for their higher signal/noise ratio, and therefore superior
detection limits. A single-collector high-resolution ICP-MS can be purchased for roughly twice
the cost of a quadrupole ICP-MS, or about $300,000. For enhanced sample throughput a
multiple-collector instrument might be purchased for about $500,000. These instruments, like
most analytical equipment, can be expected to require about 2 to10 percent of their purchase
costs in annual maintenance costs.

Thermal ionization mass spectrometers are available at a cost of $500,000. These instruments
rely not on a plasma for ionization, but rather for thermal ionization from a heated filament. They
provide more precise measurements than routine quadrupole ICP-MS but require substantially
more delicate operator involvement, leading to markedly reduced sample throughput.
Time-of-flight plasma mass spectrometers have just recently appeared on the market; they have not yet built up a historical record of performance that would permit reliable comparison with the ICP-MS equipment described above. Likewise, Fourier-transform mass spectrometers are still in the research phase and cannot yet be considered practical options for routine radiochemical analysis.

15.10.4.2 Laser

APPLICATION

Lasers can be used to excite uranium (ASTM D5174) and lanthanide complexes in solution. During or following excitation, the complex relaxes to a lower energy state by emitting photons of light that can be detected. The amount of light produced is proportional to the uranium or lanthanide element concentration.

The light emitted can be detected by fluorescence or phosphorescence. With fluorescence and phosphorescence, the detector is at right angles to Laser excitation. Fluorescence light is emitted simultaneous to the excitation.

Phosphorescence detecting differs from fluorescence in that the light emitted is not simultaneous to the excitation. This enables the light source to be pulsed and the measurement to occur when the Laser source is off. This provides improved signal to noise over fluorescence. The light signal from organic material will decay promptly, since they have a short relatively lifetime, and not be available to the detector which is gated off at this initial time. A pulsed nitrogen dye Laser can be used as the source. Other Lasers can also be used. Chloride ion and other ions may cause interferences and may need to be removed before measurement.

Kinetic phosphorimetry measures the rate of decay of the uranium or lanthanide element complex signal. Measurements are taken at fixed time intervals. In aqueous solution, the uranium or the lanthanide element is complexed to reduce quenching and increase the lifetime of the complex.

UP/DOWN TIME

Some reagents may have relatively short shelf life and need to be ordered accordingly. The life of a plasma cartridge is one to three years.
15.10.4.3 Radionuclides Analyzed By Neutron Activation

**TECHNETIUM-99**

Neutron activation analysis methods have been employed since 1972 (Foti et al. 1972a; 1972b). The method was developed and applied for the analysis of $^{99}$Tc in mixed fission products (Bate, 1979).

The method employs chemical separation of $^{99}$Tc from most fission products by a cyclohexanone extraction from a basic carbonate solution. $^{99}$Tc is stripped into water by addition of CCl$_4$ to the cyclohexanone phase and then adsorbed on an anion exchange column in a concentrated form. Neutron irradiation of the isolated $^{99}$Tc could be made in the pneumatic facility at a high flux isotope reactor (e.g., at a flux of 5x10$^{14}$ ng/cm$^2$/sec for approximately 11 seconds. Thus, after irradiation $^{99}$Tc is induced to $^{100}$Tc, which, because of its 15.8 second half-life, requires an automatic process to measure its 540 and 591 keV gamma lines.

The lower limit of detection of the analysis under these conditions is approximately 5 ng and samples up to 100 mL volume can be processed. The method has been applied successfully to reactor fuel solutions and off-gas traps containing 6.5x10$^{-4}$ to 240 µg $^{99}$Tc/mL.

**IODINE-129**

Iodine-129 can be determined by neutron activation and subsequent measurement of the 12.4 hour $^{130}$I produced by the neutron capture reaction. The method (Bate and Stokely, 1982) utilizes conventional I valence adjustments and solvent extraction to isolate the I fraction.

Chemically separated $^{129}$I is adsorbed on an anion exchange resin before being loaded for irradiation. With a neutron flux of 5x10$^{14}$ ng/cm$^2$/s for 100 seconds a lower limit of detection of 0.03 ng can be achieved.

$^{129}$I also can be determined directly by mass spectrometry (Strebin et al., 1988). The measurement limit by this technique is approximately 2 femtograms.
Special counting techniques have also been applied to the analysis of $^{129}$I. Figure 15.13 shows an efficiency plot using beta-gamma coincidence counting.

**Uranium, Thorium, and Plutonium**

Neutron Activation analysis method was employed to determine uranium in the hydrogeochemical samples from Savannah River Plants within the scope of the National Uranium Resource Evaluation Program sponsored by DOE. Uranium was determined by cyclic activation and delayed neutron counting of the $^{235}$U fission products. The method relied on the absolute activation techniques using the Savannah River Reactor Activation Facility.

Neutron Activation Analysis followed by delayed-neutron detection was commonly used for determination of $^{235}$U, $^{239}$Pu, and $^{232}$Th (Echo and Turk, 1957; Hochel, 1979; Alfassi, 1990).

### References

#### 15.11 Cited References


Nuclear Counting Instrumentation


### 15.11.2 Other Sources


Nuclear Counting Instrumentation


ATTACHMENT 15A FIELD MEASUREMENTS

15A.1 Introduction

The scope of environmental remediation work taking place across the country in the public and private sector has led to a need to improve the speed and cost-effectiveness of measurements for characterizing contaminant levels at sites and assessing the results of cleanup efforts. In particular, the time for decisions that are required during soil excavation and waste segregation should be kept short to avoid delays that tend to increase labor costs. Thus, the time it takes to collect, prepare and analyze samples can be a limiting factor. To this end, one can use mobile laboratories at the field site to reduce sample handling and transit times. However, even with these, the sheer volume of samples can overwhelm processing and analytical capacity. Therefore, measurements performed directly in the field (in situ) that do not require the collection and processing of a sample are an attractive alternative. Fundamentally, a field measurement gives the concentration of a contaminant at the same place where one might otherwise have collected a sample. In effect, the instrument is brought to the sample rather than the sample to the instrument. Frequently, the field measurement can be performed within minutes with a result obtained in what is essentially “real time.”

15A.2 Analytical Level of Measurements

Over the years, field measurements have formed an important component of standard radiological surveys. Typically, these measurements have comprised scans for gross levels of alpha or beta/gamma radiation. These types of measurements, particularly where judgment is used to evaluate a change in an instrument or audible signal, are semi-quantitative in nature and therefore would be designated at analytical level 1 under the EPA classification system used in the past or Analytical Support Laboratory (ASL) level A of the American National Standards Institute (ANSI). These levels reflect the fact that the measurement is intended for screening purposes.

However, field measurements can be performed at a higher analytical level. For example, an exposure rate measurement using a pressurized ionization chamber (PIC) is definitive for assessing the external dose rate from penetrating (gamma) radiation. In this situation, the PIC provides a direct reading of the desired measurement quantity at the actual point of interest.
Another example of a field measurement technique that has been used successfully since the 1960s is in situ gamma-ray spectrometry (ICRU, 1994). This technique provides radionuclide-specific information. In its simplest application, a spectrometer could be used to identify characteristic peaks in the energy spectrum that would point to the presence of a particular radionuclide at the measurement location. On a semi-quantitative basis, in situ spectrometry could serve as screening technique where the relative count rates—in particular spectrum peaks—are compared among measurement locations. At a higher analytical level, an appropriate calibration can be performed so that a spectrometer could be used to determine the radionuclide concentration in the media under study. Since this represents a contaminant-specific measurement where particular QA/QC checks can be made, it would be classified traditionally at the data quality objectives (DQO) analytical level 2, or ASL B.

Despite a number of successful applications of in situ spectrometry over the years, issues have arisen regarding the level of data quality that is obtained with this or any other field measurement technique for the purposes of demonstrating RCRA, CERCLA, and other regulatory compliance. In the past, field measurements by definition have not been considered to possess the quality control that needs to be established at a DQO analytical level of 4 (analogous to ASL D) in the laboratory. However, the distinction between screening level and higher level measurements is based on factors relating to data quality, which should be demonstrable. In principle, the rigorous QA/QC protocols and documentation required for analytical level 4, using EPA Contract Laboratory Program (CLP) procedures, or ANSI ASL D, could be applied to radionuclide-specific field measurements. Using field techniques at a higher analytical level is also in keeping with the latest EPA proposals for performance based measurement systems.

Typically, a projection of cost or time savings using a novel field method leads to its substitution for a more standard sampling/laboratory analysis method. In doing so, the intended applications of the field measurement method need to be established clearly. Using the DQO process, the requisite analytical level can be determined for the data that are to be collected. This analytical level should then be demonstrated through an objective judgement process whereby the data quality indicators are critically examined. Included would be those that arise when applying the DQO process (the “PARCC” parameters: precision, accuracy, representativeness, completeness, and comparability). Other related indicators or elements which can be broken out separately and which need to be addressed include documentation, instrument operating conditions, site conditions, interferences, limitations, calibration procedures, minimum detectable concentrations, reference measurements, record keeping, quality improvement, and management assessment. The following sections will provide some discussion on each of these elements as they apply to field spectrometry, the elements would generally apply to other field measurement techniques as well.
It would be expected that a demonstration of the data quality level of a field technique be performed in concert with regulatory bodies and stakeholders to obtain acceptance.

15A.3 Documentation of Methodology

A field measurement technique, like its counterpart in the laboratory, requires thorough documentation including the description of apparatus and materials, specification of personnel training/qualification level, listing of quality control checks, review of safety considerations, and issuance of non-conformance reports when necessary.

Training materials, equipment manuals, reference texts, articles from technical journals, and laboratory reports are all potential sources of background information for describing a method. It would be expected that information be extracted from these sources and a comprehensive report issued that provides the necessary background and specifics for a particular site and application. This would essentially take the form of a written procedure. For multiple applications across a site, further detail may have to be provided in project-specific plans, as the conditions under which a technique is used may vary among areas. The guidance and recommendations given by standards groups can also form a key part of documentation. Adherence to these standard procedures allows one to proceed with some confidence in the measurements process. It is expected that standards groups will increasingly devote their efforts to field measurements techniques in the future.

Individuals who will be working with the instruments and data collected need to be qualified. Educational backgrounds and necessary experience should be determined and appropriate training given for each area of work. Training and procedure manuals need to track revisions that invariably result as measurement programs progress.

Quality systems documents would include a general site-wide quality plan with specific factors like performance tests, pre- and post-operational checks, frequency of calibrations, and replicate measurements addressed in a separate method-specific quality systems section or document. Quality systems includes documenting procurement specifications for apparatus and control of materials and services such as calibration sources. Also, the turnaround time for field measurements may be important to specify not only for cost and schedule control but for limiting the time lag between measurements under changing environmental conditions.

Unforseen measurement conditions and unusual equipment malfunctions will lead to situations where doubt is cast on the validity of a field measurement. Tracking these failures will help to elucidate the problems over time and a provide a basis for corrective actions and modifications to
the procedures for future measurements. Situations where obvious bad data is collected despite
the fulfillment of QC elements will require the writing and issuance of a non-conformance report
with subsequent root cause analysis.

15A.4 Instrument Operating Conditions

Specification of instrument operating conditions is fundamental in the field as in the laboratory.
These would include power and cooling requirements as well as an acceptable range in
temperature and humidity conditions. The physical set-up of the instrument, such as a
reproducible sample-detector geometry, also should be specified. For laboratory radioactivity
counting systems, it is generally a planchet, can, bottle or similar small volume where the
distribution of activity within the sample volume is assumed to be homogeneous. For a field
measurement, the sample is in a form such as an area of ground, a storage drum, or wall.
Distances and orientation to the measured area or object need to be specified and held within
control limits.

For a field measurement, the distribution of activity within the volume of measurement should be
considered, since one does not usually have the luxury of mechanical blending as in the case of a
laboratory sample. The field of view of the detector with respect to lateral and depth
displacement within the volume under measurement needs to be established. For large volume
sources, this generally means determining the response of the instrument across all angles or
radiation incidence, not just the front face. While one cannot necessarily control the distribution
of a contaminant, the instrument response needs to be established so that the integrated signal
that it measures can be converted into a meaningful average result over the volume measured and
the sensitivity to non-homogeneous activity distributions determined.

15A.5 Site Conditions/Limitations

Preparing a site for a field measurement is analogous to preparing a sample for analysis.
Procedures need to be followed that will assure that the measurement will yield a valid result.
This might include removing obstructions and accounting for topography and ground cover such
as vegetation or surface water. The radiation absorption properties of the type of soil where
measurements are made may have to be determined beforehand depending upon the energy and
type of radiation being measured.

A significant element which should be addressed in performing field measurements is changes in
the “sample,” i.e., changes in the field conditions at the measurement point. For example,
measurements at the same location several days apart may be not be comparable if soil moisture
conditions have changed. Precipitation events would increase soil moisture, while hot, dry conditions would lead to a decrease in soil moisture.

Depending upon the instrumentation and the physical basis of the measurement, the effects of such variables as air and soil temperature, humidity, air pressure or related meteorological parameters may have to be taken into account.

Limitations should be specified for a field measurement technique. They could include site conditions such as the water content of soil, the degree and type of ground cover, the size of an area, and the estimated depth of contamination. The radionuclide mix and the concentration level could also be limiting factors.

### 15A.6 Interferences

The effects of interferences need to be assessed for proper QC in a field measurement. As compared to a laboratory setting where there is generally a controlled environment, adverse instrument effects may result from extraneous signals or electronic noise that could be produced by power line or other electromagnetic interference. Interferences in a measurement could also result from personnel—whether instrument operators or other workers—who enter into a measurement area and attenuate the measured radiation.

Whereas a laboratory counting system may employ a shield to block out background radiation, a field measurement system is exposed to ambient radiation. If significant direct or scattered (shine) radiation is present from extraneous sources, collimation or shadow shielding may be necessary. In high radiation fields, the effects of ionization in electronic components may present a problem. In this case the sensor assembly could be kept at the measurement point with the signal processing and other electronics kept at a distance.

As in the case of laboratory analysis, attention should be given to the mix of radionuclides that may be present. Interferences can result from the inability to resolve the primary energies emitted by the nuclides or because there is a high amount of secondary (scattered) radiation present.

### 15A.7 Calibration

Calibration requires that the instrument response to a known level of measured substance be determined. This generally takes the form of measuring standard reference materials or samples spiked with known quantities.
Direct calibrations using standards or spikes are usually applied to laboratory-based counting systems since only small quantities are needed for the sample volumes used. For field measurements, direct calibrations using large volume sources with a known concentration can be performed as well, although this is generally impractical and potentially expensive. In place of this, a field calibration factor for a particular source geometry and matrix composition can be derived using a two-step process. This entails determining the response to incident radiation (fluence) as a function of energy and angle (by experimental and/or theoretical means) and then calculating the fluence at the point of measurement from a given source geometry and matrix. Two-step calibration methods sometimes are applied to laboratory sample counting geometries as well.

Although a two-step process may be used for field calibrations, traceability still can exist insofar as certified point or other sources can be used in the calibration process. The calibration factor may actually represent an integrated response to a collection of sources or a single source at many different positions. In the case of a spectrometer, calibration points will need to be spaced out across the energy range of interest. Depending upon saturation effects, the calibration may also have to extend across a range in concentrations to assess the effects of signal processing dead time and pulse pile-up.

15A.8 Minimum Detectable Concentrations

Standard to any high quality measurement technique and integral to the DQO process is an a priori estimate of the detection limits of the measurement system. This needs to be done for a field measurement technique, although it may be necessary to first obtain preliminary readings in the area where measurements are to be performed. For example, the minimum detectable concentration (MDC) for a particular radionuclide will be affected by the continuum of scattered radiation present in a spectrum from other radionuclides in the soil or from sources of scattered radiation outside the area under investigation.

In some situations the sensitivity for a given count time can actually be higher for a field measurement as compared to a laboratory-based sample measurement, thus producing a lower MDC. This will result when the field detector gives a higher count rate per unit concentration because there is a far larger sample being analyzed.
15A.9 Precision

The precision of field measurements is determined with replicate measurements as in the case of laboratory measurements. To avoid potential changes in field conditions, replicate measurements can be performed sequentially with minimum time lag.

In many cases, a field measurement is a non-destructive technique. Thus, replicate measurements are easily performed. Using the results from a successive set of measurements (5 to 10), a standard deviation about the mean can be calculated. This can then be compared to the counting error for a single measurement that is based on Poisson statistics to assess precision.

Rather than perform many replicate measurements at one point, it can be more instructive to perform two or more measurements at several different points. The reproducibility can thus be judged for a variety of site conditions.

15A.10 Accuracy

Estimates of accuracy for a field measurement can be obtained through uncertainty propagation just as in the laboratory. Factors to consider include potential bias due to uncertainties in the calibration source, variations in the assumed sample/detector geometry, uncertainties in the sample matrix composition, environmental conditions, as well as the statistical counting error.

Overall system accuracy can be checked with comparisons to other techniques, or to results from an independent organization using the same technique.

15A.11 Representativeness

Representativeness refers to the degree to which a measurement reflects the condition at a location or whether a group of measurements reflects the conditions in a particular area. Generally, one desires that measurements (or samples) provide a value of a radionuclide concentration that in turn yields the best dose estimate (and thus risk) to a member of a critical group for a particular scenario. In order to achieve representativeness, a number of samples or measurements in a given area would be required in order to achieve a given confidence level or power using a statistical test.

Representativeness is affected by the heterogeneity of the contaminants in the media under investigation. Perhaps more than any other factor, field and laboratory measurements may differ at any particular measurement location due to the effects of heterogeneity. Heterogeneity can
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exist in both the lateral and depth distribution of a contaminant and can take the form of changes
in concentration across various distances: a centimeter or less, as would result from hot particles;
meters, as might occur from dumping and localized spills; and tens or hundreds of meters, as
from up-wind airborne sources. Survey designs incorporate techniques and sample/measurement
densities to accommodate these variations. The number of measurements and the standard
deviation about the mean are fundamental parameters to judge whether the mean concentration
that is measured is within a certain confidence limit. These parameters can be used to compute
the t statistic or applied to other statistical tests.

Where variations in concentration occur on a scale of tens of meters or more, it can be expected
that either field measurements or soil sampling will give similar results. It is where the variations
on the scale of a few meters or less occur that agreement between any particular pair of field
measurement and soil sample results might suffer. However, if the mean concentration in an area
should be determined, a sufficient number of measurements or samples can ultimately yield the
same average result, regardless of where the measurements or samples are taken within the area
under investigation.

Depending upon the objectives of a measurement program, a field method could inherently have
an advantage over discrete sampling. If the viewing area of a field instrument is significantly
larger than the area of a soil sample, a set of field measurement results would tend to show a
smaller standard deviation as compared to a set of soil sample data in a heterogeneous area. The
mean obtained for a given number of measurements would then be more representative of the
true mean. A wide measurement area represented by a field method could also be consistent with
the assumptions of a dose model which averages over a large area.

15A.12 Completeness

Measurement losses can occur in the field just as sample losses can occur in the lab. They result
from equipment failure, improper measurement procedures, or environmental factors beyond the
control of operators. Survey designs should incorporate allowances for sample losses by
specifying the collection of more than just the minimum number of samples needed to support a
decision.

There is somewhat of an advantage for a field technique in that QC checks can be performed at
the time of the measurement. Problems can then be immediately identified and the data rejected
on the spot. Another measurement can then be performed in place of the lost measurement.
### 15A.13 Comparability

Comparability is a critical factor that readily establishes the validity of a field technique. It can be established by performing a study in which field measurement results are compared to those given by an independent technique, such as sampling and laboratory analysis. In some situations, it may be possible to compare two different field techniques.

In performing a direct comparison study, it is important to establish that the two techniques are measuring the same thing. For instance, a technique that measures a contaminant concentration in the surface soil may compare poorly to one that is integrating down to greater depths. This situation would result where there is a non-uniform concentration depth profile of the contaminant. Where comparisons are made to soil samples, core depths can be adjusted to better match the effective viewing depth of the field measurement. The lateral distribution of the contaminant concentration across the ground could also be a factor. In this situation, compositing samples may be required to yield a better average with which to compare a field technique.

Other factors to consider for data comparability include the soil moisture and stone content of soil. Where contaminant concentrations are determined with a field technique, the value is based on the wet weight of the soil in contrast to laboratory analysis which is performed on a dry weight basis. Corrections to one of the data sets therefore need to be applied. Similarly, one should consider the effects of soil sample preparation where large stones are screened out. The concentration which is then determined is based on the activity associated with the finer particle content of the soil. If there is little or no activity in the coarser fraction, a concentration for a soil sample would be higher than that given by a field technique which has averaged in the stone content.

In place of comparing single field measurement points to single or composited samples, one can instead compare the averages of sets of field measurements to sets of soil samples over a particular size area. This would be useful to establish comparability where there is a known heterogenous distribution of the contaminant and the techniques under comparison are measuring very different areas of soil.

### 15A.14 Reference Measurements

An important QC practice in the laboratory involves the regular analysis of reference materials to confirm system calibration and performance. In practice, an analogous check can be performed for measurements in the field. A reference measurement location at a site can be designated as a field quality control station where routine, perhaps daily, measurements of the contaminants of
concern are performed. QC charts can be kept which show the results of these measurements and control limits can be specified accordingly. Unusual trends can then be identified early and corrective actions taken before unusable data is generated. Measurements at a station such as this also serve to demonstrate the effects of environmental variables such as temperature and humidity.

To further qualify a field station, intensive sampling can be performed with laboratory analyses to determine contaminant concentrations. In this situation, relatively homogeneous conditions (soil type, contaminant concentration) would make the comparison more favorable and help to trace any bias between measurement methods that might be observed.

In addition to reference materials, the analysis of blanks is a regular feature of laboratory-based counting systems. This establishes that contamination of equipment and materials has not occurred. Similar contamination can occur to field instrumentation such as wind blown soil particles in crevices, encrusted mud on the underside of equipment, or soil plugs in tripod legs. For a field measurement technique, it may be possible to check self-contamination by performing measurements in a background area where the contaminant in the soil is essentially zero. If the contaminant is present in background, such as $^{137}$Cs from nuclear weapons fallout, an offsite area at least can serve to establish a regional baseline measurement. As a standard measure of precaution, routine scanning of equipment can be performed with friskers, especially after work in highly contaminated areas.

15A.15 Record Keeping

Field personnel need to use log sheets or books to record necessary information about the site conditions, measurement parameters, and data storage. In place of chain of custody forms for samples, analogous records may be required for data printouts or electronic files of results (spectral data) obtained in the field as they pass through different levels in the organization (data entry, data analysis, validation, etc.).

Maintenance logs or files on specific pieces of equipment need to be kept. Factory repairs or in-house replacement of components should be noted as any changes to an instrument are likely to require recalibration. Equipment and component failures should also be tracked.

15A.16 Quality Improvement

Operating experience generally leads to fuller knowledge of instrument performance and characteristics as well as better recognition of precursors to problems. Based on control chart
records, observations and correlations with other factors associated with a measurement, breakdown and repair logs, and the information contained in any non-conformance report, procedures can be modified to improve data recovery and usability. In time, the net effect of changes incorporated in standard operating procedures will lead to improvements in performance tests. Along with the identification of limiting factors and the development of solutions, it may be possible to justify raising the analytical level of the measurement based on the quality control indicators.

15A.17 Management Assessment

In addition to the quality control elements in place when a field technique is demonstrated initially, systems need to be in place to insure that data quality is maintained in subsequent measurements once the technique is used routinely. Deployment of a field methodology on a broader scope generally entails use by non-experts, i.e., individuals not associated with the development or implementation of an instrument. For this reason, internal assessments may be needed in the form of independent oversight (audits). The data verification and validation process can be used to insure the fulfillment of QC checks. Ultimately, data may have to be reviewed and approved by individuals who have expertise with the measurement system.

15A.18 Combined Laboratory and Field Measurements

Laboratory and field measurement techniques are not mutually exclusive. They can frequently be used in concert to achieve better and more cost-effective radiological surveys. A likely combination would be reliance on field methods which are faster with the laboratory method serving as a QC check. Appropriate ratios in the number of field to lab measurements would have to be established based on expert judgement and by reviewing the data quality objectives. The ratio could vary from area to area within a site depending upon the situation and the presence of complicating factors.

15A.19 References