

16 DATA ACQUISITION, REDUCTION, AND REPORTING FOR NUCLEAR-COUNTING INSTRUMENTATION

16.1 Introduction

This chapter provides information and guidance, primarily for laboratory personnel, on data acquisition, reduction, and reporting for nuclear-counting instrumentation processes. Its intent is to provide an understanding of the many operational parameters that should be addressed in order that the data developed and reported are compliant with project planning documents (Chapter 4), considered valid (Chapter 8, *Radiochemical Data Verification and Validation*), and usable for their intended purposes (Chapter 9, *Data Quality Assessment*). These processes are all linked and each is dependent upon the results of its predecessor. The material presented is intended to provide an overview of the processes that are used in all radiochemistry laboratories, but are by no means performed in the same way in all laboratories.

In this chapter, data acquisition refers to the results produced by nuclear-counting instrumentation. This chapter will provide guidance for laboratory personnel on selecting and applying the operational parameters related to instrumentation and the determination of the radioactivity contained in the test source.¹ Parameters that are applicable to counting for essentially all radiation detection instrumentation are discussed in Section 16.2, and those that are specific to a given type of instrumentation are covered in the appropriate section describing that instrument. Detailed descriptions of the instruments discussed in this chapter are provided in Chapter 15, *Quantification of Radionuclides*.

Once test sources have been prepared and counted using laboratory measurement instruments (Chapter 15), the basic information generated by the instrument should be processed and reduced to data that can be reviewed, verified, validated, and interpreted in accordance with project planning documents and analytical statements of work (SOWs; also see Chapters 5, *Obtaining Laboratory Services*, and 7, *Evaluating Methods and Laboratories*). Data reduction is primarily mathematical in nature while data reporting involves the presentation of the results of the data acquisition and reduction

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¹ The term “test source” means the radioactive material prepared to be introduced into a measurement instrument, and “laboratory sample” means the material received by the laboratory for analysis. Thus, a test source is prepared from laboratory sample material in order to determine its radioactive constituents. A “calibration source” is a source prepared for the purpose of calibrating instruments.

processes and nonmathematical information necessary to interpret the data (e.g., sample identification and method of analysis).

Data reduction may be as simple as a division of the counts by the counting time, the sample aliquant weight or volume, and the detector efficiency, thereby producing the radionuclide concentration. On the other hand, it may also require more complicated processing such as the fitting of an analytical function, or the unfolding of a differential spectrum (Tsoulfanidis, 1983). In any case, the reduction process should continue by calculating the combined standard uncertainty (Chapter 19, *Measurement Uncertainty*).

The output of some laboratory instruments is highly simplistic and consists only of the number of nuclear decay events recorded by the detector in the time interval allocated for the measurement. An example of this might be a proportional counter whose only output is from an electronic scaler, and the available data consist of total counts or count rate. On the other extreme, some laboratory counting instruments with computer components produce outputs consisting of radionuclide concentration, uncertainty, and other information. Examples of these types of data reducing instruments are alpha- and gamma-spectrometry and liquid-scintillation systems.

ANSI N42.23 contains an outline of a minimal data report. Most project-specific planning documents or analytical SOWs require that the radiochemical data produced by laboratories be submitted in a specific format and form (i.e., electronic or hard copy, or both). In some cases, the requirements are minimal and may consist of a data report that gives only the sample identifier information, accompanied by the radionuclide concentration and its associated uncertainty. Many projects require much more supporting information, primarily to assist in the data validation process. Support material can include information on calibration, background determination, sample processing, sample receipt, quality-control sample performance, raw-counting data, and chain-of-custody records.

This chapter gives an overview of data acquisition, reduction, and reporting in radiochemical laboratories. The material presented is intended to be descriptive rather than prescriptive, since these processes vary greatly between laboratories; depending upon the equipment, personnel, project requirements, and the methods and analyses being performed.

16.2 Data Acquisition

Data acquisition in this context refers to the process of collecting the basic information produced by nuclear-counting instrumentation. These data may be produced in hard copy or electronic format, or visually displayed for the operator to record. As previously stated, this can be simply the number of counts detected by the instrument within the allotted counting time or as conclusive as the identification of the radionuclides contained in the sample along with their concentrations and associated uncertainties.

Following generation, data requiring further processing may be transferred electronically or manually to the next data-reduction step. Electronic transfer should be employed as often as possible, as long as the software process has been verified and validated to perform correctly in this function. Software responsible for electronic data transfer should be validated and verified initially, and any changes verified and validated. A manual recheck of some portion of the data analysis should be performed on a routine basis (e.g., annually).

The reliability of the data generated also depends upon the proper operation of the instrumentation and the associated data reduction programs. Data quality further depends upon the correct input of associated information by laboratory personnel.

16.2.1 Generic Counting Parameter Selection

Instrument operators have choices, provided by instrument manufacturers, in the setup and operation of nuclear counting instruments. These selections can affect the quality and applicability of the data. Some selections can be made on a one-time basis and left unadjusted for the processing of all samples and others require the operator to reevaluate the settings, possibly for each test source counted. In some cases adjustments can be made following counting during the processing of the derived information. Some adjustments can only be made before counting or by extending the counting time. In making the proper selection, there are some overall considerations relative to the project requirements, as specified in project planning documents or in the analytical SOW. Other operator decisions depend on the nature of the test source itself. Caution should be exercised when changing operational parameters so that the calibrations (counting efficiency, energy, self absorption, etc.) performed on the instrument remain valid. For example, changing the source container or holder may affect the counting efficiency and/or background. Determining the appropriate operating conditions requires that the operator have a thorough understanding of the counting process and the instruments and their operation for the production of valid and useable data. In addition, the operator should be cognizant of the measurement quality objectives (MQOs) that have been established.

Some of the factors that affect operational parameter selection are related to project requirements. Planning documents and the analytical SOW may specify the limits on measurement uncertainty and detection capability. In order to achieve compliance with the limits, adjustments to instrument operating parameters (e.g., count times) may be required for some or all the samples received. The number of samples received during a time period may make it mandatory for adjustments to be made in order to meet these requirements while complying with project-defined turnaround times.

Factors that may affect the selection of operational parameters include:

- Project and External
 - project requirements for uncertainty, detection capability, and quantification capability

- laboratory backlog, radiological holding time, and sample turnaround times
- Sample Characteristics
 - expected sample radionuclide concentration
 - interfering radionuclides
 - interfering stable constituents (e.g., liquid scintillation counting quenching)
 - amount of sample available
 - physical characteristics of the test source (e.g., density)
 - half-life of the radionuclide of interest
- Analytical Process
 - chemical separation process leading to test-source generation
- Instrumentation
 - instrument adjustments available and their limits
 - conditions and limits of an instrument's calibration
 - time availability of instruments
 - counting efficiency
 - calibration geometries available

Taking into consideration the above, the operator has control over and should select certain parameters for all radiation measurements. The selection of the basic parameters should be carefully planned in advance to assure that the project requirements are met. The laboratory's selection of parameters during the planning process may require alteration as the process of sample analysis is actually taking place due to unavoidable changes in the samples and sample characteristics throughout the duration of the study.

16.2.1.1 Counting Duration

The standard uncertainty of a measurement with total number of observed counts, N , using Poisson counting statistics, equals the square root of N (as further explained in Chapter 19). The relative fractional uncertainty of the measurement of N is then $1/\sqrt{N}$. The expected value of N is proportional to the length of the counting period; so, increasing the counting duration, which is a controllable factor, can reduce the relative uncertainty of the measured counts. The analyst then should select counting durations for the sample and the blank that are sufficient to meet the project objectives for detection capability and method uncertainty. An alternative to selecting the counting duration, available on many radiation counting instruments, is to count until a preset number of counts is obtained.

Note that the overall measurement uncertainty for the final analytical result usually depends on many factors besides the counting uncertainty; so there is a limit to the improvement that can be made by adjusting counting times alone.

16.2.1.2 Counting Geometry

The counting efficiency of a radiation detector depends upon (among other things) the geometry of the source and detector arrangement, i.e., the solid angle subtended at the detector by the source (see Chapter 15, *Quantification of Radionuclides*). A given radiation detector may have the counting efficiency established for several geometries. The geometry selected among those available may depend upon the amount of sample available, the quantification requirements for the analysis, the radionuclide concentration in the sample, the dictates of the radioanalytical method, the physical characteristics of the sample, the nature and energy of the decay process, and the characteristics of the detector.

The choices to be made relative to geometry selection are usually the type of test-source container, the source mounting, and the source-to-detector distance. Choices are to be made among those for which the detector has an established efficiency calibration.

16.2.1.3 Software

The use of properly developed and documented computer software for data acquisition and reduction can enhance the quality of laboratory data. Guidance on software documentation can be found in EPA (1995). Caution should be exercised in the selection and use of undocumented programs and those which may not have been tested in laboratories performing analyses similar to those for which MARLAP has been developed. For example, a spectral analysis program may accurately identify and quantify the radionuclides in test sources containing higher levels of radioactivity (which produce spectra with well-defined peaks, easily distinguishable from background) but may be inaccurate for samples with environmental radionuclide levels.

When selecting software, one should thoroughly review the data reduction algorithms. The user should not blindly accept the notion that all software performs the calculations in an appropriate manner without this review. When evaluating software, it is often helpful to review the software manual, particularly in regard to the algorithms used in the calculations. While it may not be necessary that the user understand in detail all the calculations performed by highly complex software programs, the user should understand the overall scheme of analysis and reduction in order to assure data meet quality objectives and reporting requirements. This understanding is also beneficial in assuring that user-defined parameters are properly selected.

The output of some instruments is very basic, consisting primarily of counting data (total counts or count rate). These data should be manipulated by external systems to convert them to the form required by planning documents. The external system that performs the calculations may be a calculator or a computer with the appropriate software to reduce the data to usable terms. In either case, additional information relative to the processing of the sample should be input along with the counting data (counting time, total counts, and background counts). This information may include laboratory sample identifier (ID), collection date, sample mass or volume processed, instrument counting efficiency, and chemical yield.

For computer (processor) based systems, some of this information is generated and processed internally and the remainder is manually entered or electronically transferred from the Laboratory Information Management System (LIMS) or some other adjunct system where it has previously been stored. It is becoming increasingly common for much or all of this adjunct information to be transferred to the counting instrument by reading a bar code affixed to the test source to be counted. In this manner, the information that has previously been entered into a LIMS is electronically transferred to the counting instrument. For hand calculations, these data are simply entered into the calculations.

The software data reduction and reporting functions should be verified to perform as expected. For example:

- Manual calculations and software calculations performed on the same raw data should produce the same analytical results; and
- Calculation of activity using secondary/tertiary gamma rays of a radionuclide should consistently validate the activity determined from the primary gamma ray.

16.2.2 Basic Data Reduction Calculations

The equations used for data reduction depend on the analytical methods used. The following equations are provided as examples to illustrate the basic principles involved in data reduction.

Following counting, the radionuclide concentration may be calculated:

$$R_C = \frac{C_G - C_B}{\epsilon \cdot V \cdot Y \cdot K_C \cdot e^{-\lambda t_1}} \quad (16.1)$$

where:

- R_C = radionuclide concentration at a reference time (i.e., time of collection) (Bq/L or Bq/g)
- C_G = gross counting rate (source + background) (cps)
- C_B = counting rate of the blank (cps)
- ϵ = detector efficiency for the radionuclide being measured (cps or Bq)
- V = volume or mass analyzed (L or g)
- Y = chemical yield (when appropriate)
- e = base of natural logarithm
- λ = radioactive decay constant for the radionuclide (reciprocal time units)
- t_1 = time lapse from sample collections to beginning of source count (units consistent with λ)
- K_C = correction for decay during counting and is:

$$K_C = \frac{1 - e^{-\lambda t_C}}{\lambda t_C} \quad (16.2)$$

where:

t_C = clock time (total time during which counting occurs) of counting (units consistent with λ). Clock time, live time, and dead time are discussed below.

Equation 16.1 calculates the radionuclide concentration at the time of sample collection. It compensates for the fact that short-lived radionuclides may experience significant reduction in activity during counting, when the counting duration is a significant fraction of the half-life.² For long-lived radionuclides ($t_{1/2} > 100$ times the counting time), the term K_C approaches unity and may be ignored. The efficiency used in this equation may be obtained from the specific radionuclide whose concentration, R_C , is to be determined or it may be obtained from an efficiency curve that plots detector efficiency against energy. In the latter case, the emission probability per decay event, E_e , (also called “abundance,” “percent abundance,” or “branching ratio”) of the particle or photon being counted must be considered. This is required because the energy dependent efficiency, ϵ_e , is developed in terms of the fraction of particles or photons detected divided by the number emitted at that energy. Thus, if the radionuclide emission being determined during the counting of a test source has an abundance less than 100 percent, an adjustment should be made to Equation 16.1, as shown in Equation 16.3:

$$R_C = \frac{C_G - C_B}{E_e \cdot \epsilon_e \cdot V \cdot Y \cdot K_C \cdot e^{-\lambda t_1}} \quad (16.3)$$

Most modern instrument systems contain software to perform data manipulations that convert basic counting information to a form that can be compared to the project data quality objectives, or at least to begin or promote this process. Certain sample-specific information should be manually entered or transferred to the system electronically in order to perform the necessary calculations.

“Live time” is the time period that the analyst chooses to count the sample. “Dead time” is the period that the counting system is unable to process multiple detection events within the resolving time of the analog-to-digital converter (ADC) plus storage in the correct memory channel. All counting systems have some dead time. Live time plus dead time is called “clock time.” For environmental samples, or when using gamma spectroscopy or liquid scintillation

² If several half-lives of the radionuclide elapse between sampling and analysis, decay-correcting the result to the time of sampling increases both the measured concentration and its uncertainty. When a result that is statistically indistinguishable from zero is decay-corrected in this manner, the corrected result may be positive, negative, or zero, but the magnitude is often so large that it causes concern to data users. See Attachment 14A, “Radioactive Decay and Equilibrium.”

systems, dead time is usually negligible, because decay events (even with multiple radionuclides present) randomly occur far enough apart (Canberra, 1993). In these cases, the live time is the same as elapsed time (elapsed time sometimes is referred to as “real” or “clock” time). However, as the sample activity increases, the probability of two decay events happening within a short time of each other also increases. When the first event is being processed by the ADC, the ADC will not accept another pulse until the output of the ADC is stored in the correct memory channel (the period of dead time). If a second event occurs during the detection and count-system analysis of the first event, the second event is not counted. Some counting systems compensate for this by using the “preset time,” which does not advance during this detector processing period. (Preset time thus is synonymous with live time.) Many systems have meters that indicate percent dead time. This can be expressed by Equation 16.4.

$$\% \text{ Dead Time} = \frac{\text{Clock Time} - \text{Live Time}}{\text{Live Time}} \times 100 \quad (16.4)$$

Increasing the live time by this percentage yields the clock time. Although clock time and live time are very close, clock time should always be used in ingrowth or decay calculations, especially with radionuclides whose half-lives are significant with respect to the counting interval.

The best method of compensating for high dead time samples is to either dilute the sample or use less of it for the analysis. High dead times can cause other problems with the counting systems such as peak shaping and signal recognition, which can affect results.

16.3 Data Reduction on Spectrometry Systems

Software is available for resolving alpha, gamma (including X-rays), and liquid scintillation spectra and for performing the attendant functions such as calibration, energy alignment, background acquisition and subtraction, and quality control (QC) functions.

Spectroscopic analysis for alpha particles and gamma rays is performed to identify and quantify radionuclides in samples. Since these emissions occur at discrete energies, spectrometry is useful for these purposes and can be applied to the analysis of a wide range of radionuclides. Energy spectra are produced when a detector absorbs a particle or photon and produces a signal that is proportional to the energy absorbed. The resulting signal is digitized by an analog-to-digital converter and processed by a multichannel analyzer. A differential spectrum is produced, where the number of events within an incremental energy, ΔE , is recorded on the y axis and the energy is represented on the x axis (Tsoulfanidis, 1983). In this way, radionuclides can be identified by the characteristic energies of their emissions and quantified because the area under the full energy peak is proportional to the emission rate of the source being analyzed and to the count time.

The spectra for alpha and gamma emitters are quite different, due to the differences in the way these two types of radiation interact with matter in transferring their energy to the detector material. The process of resolving the spectra into its contributing components is referred to as spectral analysis (NCRP, 1978) and unfolding (Tsoulfanidis, 1983). Computer programs for analyzing alpha and gamma spectra are available from several sources (Decker and Sanderson, 1992). A method of performance testing of gamma analysis software is given in ANSI N42.14.

16.3.1 Gamma-Ray Spectrometry

Gamma-ray spectrometry on environmental samples requires the use of gamma spectral analysis software for any reasonable degree of accuracy and detection capability. (Reference to gamma rays and their detection in this context also includes X-rays from radionuclide decay.) This is due to the potentially large number of photopeaks to resolve, the low level of radioactivity in most environmental samples, and the relatively low detection limits and stringent QC requirements of most project-specific planning documents. Spectral analysis by manual techniques is only practical when the number of radionuclides is limited and the contributing radionuclides are predictable. An example is the analysis of milk samples for gamma-emitting radionuclides, where the milk production process in the cow restricts the number of radionuclides in the milk product (Hagee et al., 1960; USPHS, 1967).

Gamma rays interact with matter in three ways: by photoelectric effect, Compton scattering, or pair production (Tsoulfanidis, 1983). These interactions within a gamma detector (usually high-purity germanium or sodium iodide; see Chapter 15) result in varying amounts of the gamma-ray energy being absorbed. Only one—the photoelectric—results in the total energy being absorbed in a single interaction. The photopeaks in Figure 16.1 result from the processing of the detector signal through the linear circuitry and the multichannel analyzer.

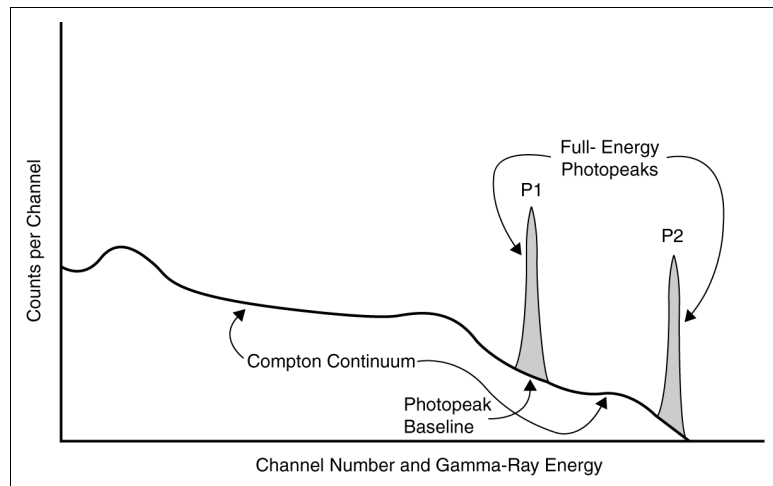


FIGURE 16.1 — Gamma-ray spectrum

As can be seen in the figure, a lower-energy photopeak (P1) may be displaced upward by combining with the accumulated counts from the Compton continuum, generated from other possible higher-energy photopeaks (P2) and background radiation. Each photopeak has a basic Gaussian shape (Gilmore and Hemingway, 1995). It may be described with the baseline counts removed from each peak channel (Quittner, 1972) by:

$$f(x) = A e^{-(x-p)^2 / 2\sigma^2} \quad (16.5)$$

where:

- $f(x)$ = the expected number of counts in any channel x
- x = the channel number
- A = the peak amplitude (counts in the centroid channel)
- p = the peak centroid channel
- σ = the standard deviation of the Gaussian peak

(The width of the peak is related to the full-width at half-maximum [FWHM] of the detector, Γ , where $\Gamma = 2.355 \sigma$. The area under the peak is $N = 1.064 A \Gamma$.)

The photopeak is the key element in gamma-ray spectrometry in that its location on the energy axis provides a means for radionuclide identification, and the area of the photopeak is proportional to the number of photoelectric events detected. This becomes the basis for radionuclide identification and quantification.

The fundamental purposes of gamma-ray computer-based spectral analysis programs are to identify the photopeaks in a spectrum and to measure the true area under the photopeaks. It should do this in the presence of natural background, a potentially large number of sometimes overlapping photopeaks, and a great number of Compton-scattering events. Once these initial tasks have been performed, the computer program uses this information to determine the radionuclide mix that contributed the complex spectrum and the individual concentrations in the sample being analyzed.

Most computer programs for gamma-spectral analysis are provided by equipment manufacturers, although some are supplied by independent providers. There are significant differences in the structure of the programs. However, they all perform similar functions, which are given below and illustrated in Figure 16.2.

16.3.1.1 Peak Search or Identification

There are two basic methods of gamma spectral analysis. The first method is to allow the analysis software to determine the existence of the peaks and their energy. The second method is often referred to as a “library directed” search, where the operator identifies the peak energy locations, e.g., regions of interest, to be searched for discernable peaks. The latter method may be more sensitive (Gilmore and Hemingway, 1995) but, taken alone, will fail to identify and report unspecified radionuclides. If the confirmation of the existence of a particular radionuclide is required, the second method should be employed. Most software programs allow either approach to be activated and used for each analysis. If only the regions of interest technique is used to assess the concentration of a radionuclide, it is still important to assess the presence of other radionuclides. For example, when determining ^{134}Cs (at the 604.7 keV peak), a false positive

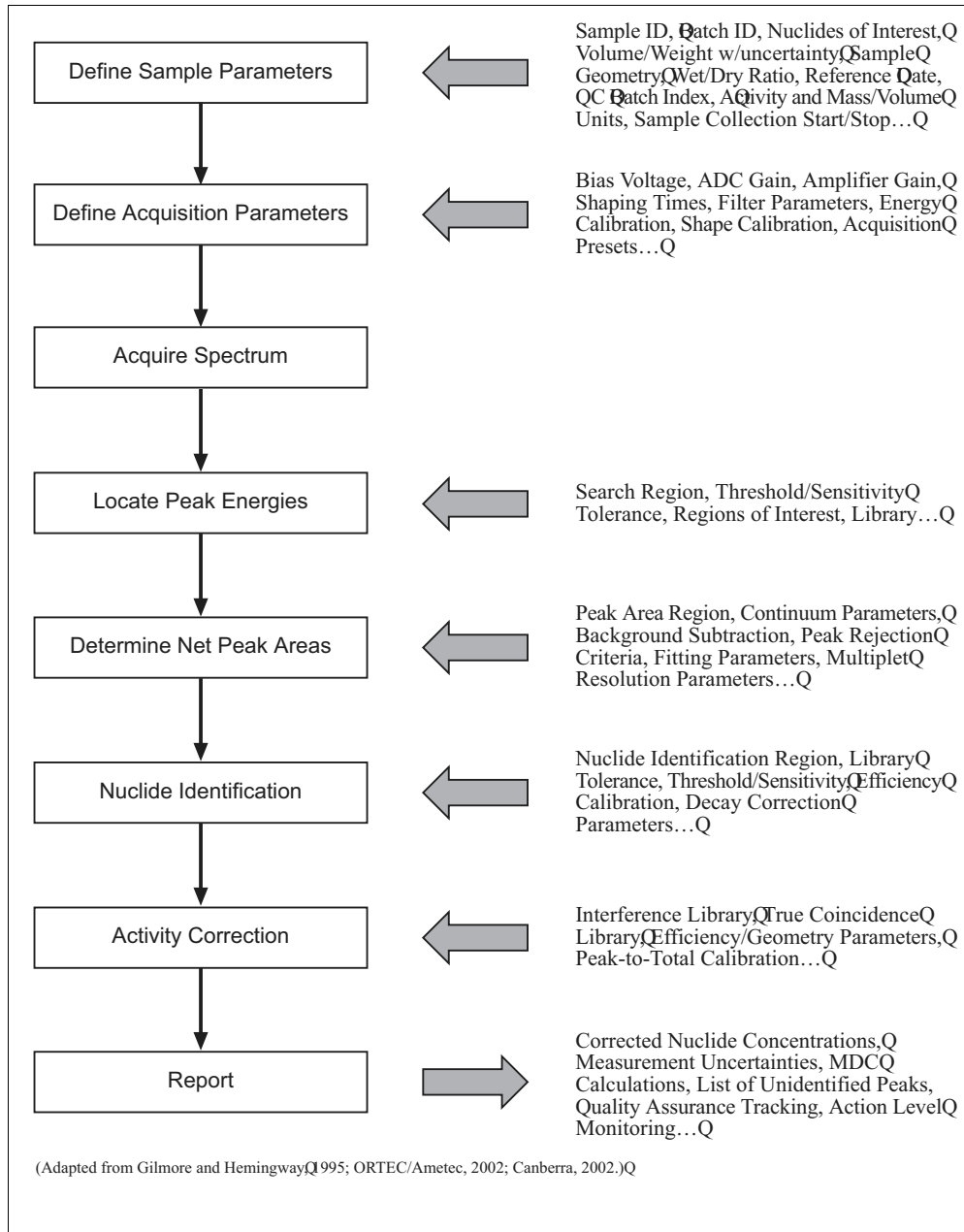


FIGURE 16.2 — Gamma-ray analysis flow chart and input parameters

result or high bias might be realized for the ^{134}Cs , if ^{125}Sb (606.6 keV) were present.

A most important function performed by an analysis program is the identification of true photopeaks. In the programs available, this is achieved in one of the four ways discussed below. Many spectral analysis programs allow the operator to select among two or more of the four methods for peak identification. Selection of the most accurate and sensitive method depends on the radionuclides present in the source, detection capability requirements for individual

radionuclides, the number of radionuclides present, the nature of the background spectrum, the degree to which the radionuclide mix can be predicted, and the activities of the radionuclides. The selection of a particular peak search method can be determined by experience with similar sample types and past performance, particularly on performance evaluation (known) samples.

REGIONS OF INTEREST (ROI) METHOD

This is the simplest form of peak identification, but can only be used when the radionuclides present in the sample are known and when the analysis system has been compensated for gain drift. ROI analysis involves the establishment of predetermined energy regions, at least one for each radionuclide present. Once the spectrum has been acquired, the number of counts in each region is summed after subtracting the photopeak baseline (Figure 16.1). This method of spectral analysis may be more applicable to alpha rather than gamma-ray spectrometry.

GAUSSIAN FUNCTION DERIVATIVE METHOD

As previously stated, the photopeak has a basic Gaussian shape; in reality it is collected, stored, and presented in a histogram format. Mathematically, it is represented by an exponential on the low- and high-energy sides and Gaussian in the middle. The most widely used peak identification technique was proposed by Mariscotti (1967). This technique uses derivatives of the Gaussian function to assess the presence of a photopeak. For most low-level radioactivity, this peak search method may provide the best peak detection capability with the fewest false peak identifications or omissions of true peaks.

CHANNEL DIFFERENTIAL METHOD

This method searches for a number of channels where the counts are significantly greater than the preceding channels, and then looks for the expected decrease in counts corresponding to the backside of the prospective photopeak. This method works relatively well for large, well-defined peaks, but is limited for poorly defined peaks with counts barely above the background baseline of the peak (Gilmore and Hemingway, 1995).

CORRELATION METHOD

In this method, a search function is scanned across the spectrum. Each channel count, over the width of the search function, is multiplied by the corresponding value of the search function. The sum of these products is then made a point on a correlation spectrum. A correction for the baseline contribution leaves only positive counts within a photopeak. Although the scan function is normally Gaussian in form, other forms may be applied (Gilmore and Hemingway, 1995).

Spectral analysis programs usually have some user selected peak acceptance criteria. The acceptance criteria may be based on peak shape, width uncertainty, or the number of standard

deviations above the background to be subtracted. Care is required in selection of the values for these acceptance criteria. If the values are too high, valid photopeaks remain undetected. If the values selected are too low, radionuclides may be reported that are not present in the samples. Knowledge of the sample origin and experience with using the analysis program on similar samples to those being processed is useful in establishing values for these user-selected parameters. Peak searches may be standard or directed (Canberra, 1994). In a standard search, all identified peaks are assigned to a radionuclide according to the nuclide energy values contained in a radionuclide energy library. In a directed search, the user specifies the energies and radionuclides over which the search is performed. If reporting a specific radionuclide is required, the directed search is appropriate; however, some radionuclides could go unreported if only a directed search is performed. Nuclides with multiple gamma rays may have several (but not all) gamma rays identified in the library. Depending upon user-selected criteria in the software, peak matches to library listings may be for a single line of the nuclide or multiple lines. An example is ^{60}Co , with gamma rays of equal intensity at 1,173 and 1,332 keV. A software option may be selected so that both gamma rays must be found for the positive identification of the ^{60}Co radionuclide. In this case, if only one of the peaks was found (as can occur for very low radionuclide concentrations), ^{60}Co would not be listed in the final analysis report.

In order to identify gamma peaks and radionuclides correctly, the user should select a radionuclide-energy library corresponding to the correct sample matrix or to those radionuclides in the sample based on the origin of the sample (e.g., SOW- or project-identified radionuclide list, soil samples, power plant effluent samples, etc.). For example, a radionuclide energy library for soil samples will include the gamma-ray energies associated with the radionuclides in the naturally occurring uranium and thorium decay chains. However, using this library to evaluate spectra for reactor coolant water samples having short-lived fission or activation products would be inappropriate, because some observed gamma peaks may not be identified and other observed gamma peaks may be misidentified as naturally occurring radionuclides. A radionuclide energy library may also be tailored according to the half-life of the expected radionuclides. Use of such a library avoids identification of a radionuclide whose half life would prohibit its presence in a sample.

16.3.1.2 Singlet/Multiplet Peaks

A peak is referred to as a singlet or multiplet according to whether it is composed of a single photopeak or multiple photopeaks, respectively. "Deconvolution" is the term given to the process of resolving a multiplet into its components (Gilmore and Hemingway, 1995). The ability of a spectral analysis program to perform this function may well be the deciding point for its selection. It is particularly important if the laboratory has analyses in which one of the critical radionuclides has only one gamma ray whose energy is very near to that of another radionuclide expected to be present in all or most samples.

There are three primary ways that programs deal with the problem of resolving multiplets. The

first method is with a deconvolution algorithm, which is based on the peak shape being the composite of multiplet Gaussian distributions. The second method uses the gamma-ray library to anticipate where peaks occur within a multiplet. The disadvantage of the first is in dealing with small ill-defined peaks and the second cannot, of course, resolve peaks not included in the library. The third method, peak stripping, again depends on defining all radionuclides whose gamma rays contribute to the multiplet. In peak stripping, one of the interfering gamma ray's contribution is subtracted from the multiplet area by using another of its gamma rays to estimate the peak shape and size in the multiplet area. The remaining peak is, presumably, that of the interfered radionuclide, which can then be identified and quantified. This method requires that one of the interfering radionuclides have a second gamma emission that identifies and tentatively, for the purpose of removing its contribution, quantifies it.

In some cases, the uncertainty of multiplet deconvolution can be avoided by selecting energies of gamma rays that are not interfered with, even though they may have lower abundances. The increase in uncertainty due to the lower number of accumulated counts may well overcome the uncertainty of deconvolution (Gilmore and Hemingway, 1995).

16.3.1.3 Definition of Peak Centroid and Energy

Once a peak has been detected, the centroid of the peak will be defined, since it will rarely be located at exactly a whole channel number. The centroid will be used to represent the gamma-ray energy and should be calculated to the fraction of a channel. An algorithm used to calculate the centroid value may be expressed as (Gilmore and Hemingway, 1995):

$$\text{Centroid} = \frac{\sum C_i i}{\sum C_i} \quad (16.6)$$

where C_i is the number of counts in the i^{th} channel.

In order to assign a gamma-ray energy value to the peak centroid channel position, the analysis program refers to a previously established energy calibration file. The detector's response to the full range of gamma energies should be established by counting one or more sources having a number of well-defined gamma rays over the range of energies emitted by the radionuclides in the calibration source. This calibration source most often is a "mixed-nuclide source" with certified emission rates, so that it also may be used for an efficiency calibration. The mixed-nuclide source is counted on the detector, being sure to accumulate sufficient counts in the peaks to obtain good statistical precision, and an energy-versus-channel relationship is established. The operator will be required to provide information on the peaks to be used and their exact energies.

With modern spectrometry systems, the relationship between energy and channel number is nearly linear. Both linear and quadratic fits have been included in available spectral analysis programs.

16.3.1.4 Peak Width Determination

In order to calculate the area under the peak, an estimate of the peak width is required, unless the analysis program is operating in the region-of-interest mode. The width of a photopeak is normally quoted in terms of its FWHM (see also Section 18.5.3.2, “Peak Resolution and Tailing”). For a discussion of peak width (resolution) and the factors affecting it, see Chapter 15.

There are several ways to determine the peak boundary. These are:

- (1) A Gaussian shape is assumed and some number of standard deviations (2 or 3) are allowed on each side of the peak centroid.
- (2) A standard width for each peak, based on its energy, is used.
- (3) A five-point moving average is used to determine a minimum on each side of the peak, which is set as the peak limits.

Each method has strengths and weaknesses, but all struggle with ill-defined (small number of counts) peaks. Once the peak limits are defined, determining the area under the peak is accomplished by summing the counts per channel for the channels contained in the peak and subtracting the baseline (Figure 16.1).

The determination of FWHM requires an assumption of peak shape, and for gamma-ray spectroscopy, the peak shape is assumed to be a Gaussian function. In addition, the peak width increases with the energy of the gamma ray, so some function should be defined for the analysis program to determine the width based on the energy of the peak. This relationship, in practice, is found to be nearly linear (Gilmore and Hemingway, 1995) and described by:

$$w = a + bE \quad (16.7)$$

where:

- w = width of the peak
- E = the energy
- a, b = empirical constants

For spectra developed by high-purity germanium semiconductors (HPGe) and alpha solid state detectors, it may be more appropriate to assume a peak shape that is a modification of the Gaussian function to allow for the low energy tailing observed in these spectra. This type of tailing is illustrated in Figure 16.3. Some spectroscopy programs have algorithms to fit peaks with lower energy tailing.

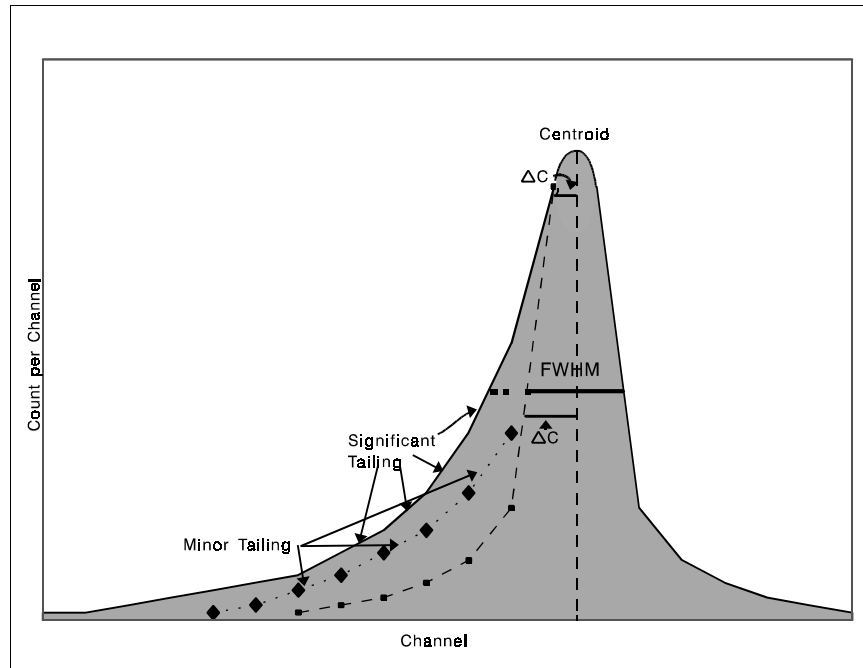


FIGURE 16.3 — Low-energy tailing

When the “tailing” peak fit option is selected, the software algorithm for peak fitting changes from the pure Gaussian form to a dual fit. The channels in the peak not affected by the tailing are included in the Gaussian fit (Equation 16.8), and those that are affected by tailing are modified according to Equation 16.9 (Koskelo et al., 1996):

$$y(x) = \begin{cases} A e^{-\frac{(x-p)^2}{2\sigma^2}}, & x \geq p - \Delta C & (16.8) \\ A e^{-\frac{\Delta C(2x-2p+\Delta C)}{2\sigma^2}}, & x < p - \Delta C & (16.9) \end{cases}$$

where:

- x = the channel number
- A = the peak amplitude
- p = the peak centroid channel
- ΔC = the tailing factor (the distance from the centroid to the point where the tailing joins the Gaussian peak)
- σ = the standard deviation of the Gaussian peak ($\approx \text{FWHM} / 2.355$)

It should be noted that tailing may have many causes:

- Electronics temperature changes due to room temperature variation;

- Interferences from other gamma rays whose energies are <1 FWHM from the centroid;
- Phonic interference from vibration (e.g., a turbine);
- Detector degradation; or
- Detector temperature changes due to liquid nitrogen level.

These concerns should be corrected rather than trying to compensate for them mathematically using a correction factor.

16.3.1.5 Peak Area Determination

For single peaks sitting on a Compton continuum, two methods of peak area determination are available. The less complex method is the addition (integration) of the number of counts per channel in each of the channels considered to be within the peak limits, and subtracting the natural background and Compton contribution to those same channels (Baedeker, 1971; Loska, 1988). However, this is rarely simple since the photopeak is usually offset by a baseline continuum whose contribution is not easily determined. While the background may be subtracted by the spectrometry program, the Compton continuum will be estimated by the software and then subtracted. This estimation is often based on the number of counts per channel in those channels immediately above and below the photopeak region as shown in Figure 16.4 (after Gilmore and Hemingway, 1995).

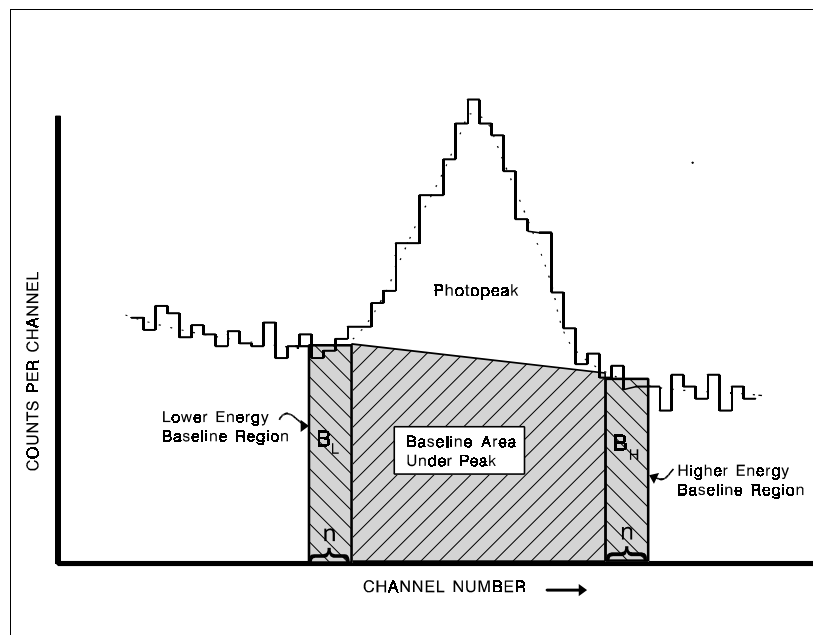


FIGURE 16.4 — Photopeak baseline continuum

The baseline contribution is then estimated as:

$$B = \frac{N}{2n}(B_L + B_H) \quad (16.11)$$

where:

- B = the number of counts attributed to the baseline
- N = number of channels in the peak
- n = the number of baseline channels considered on each side of the peak for calculating B_L and B_H
- B_L = the sum of the number of counts in the baseline region on the low-energy side
- B_H = the sum of the number of counts in the baseline region on the high-energy side

In practice, the baseline continuum appears to have a step beneath the peak (Gilmore and Hemingway, 1995), as illustrated in Figure 16.5.

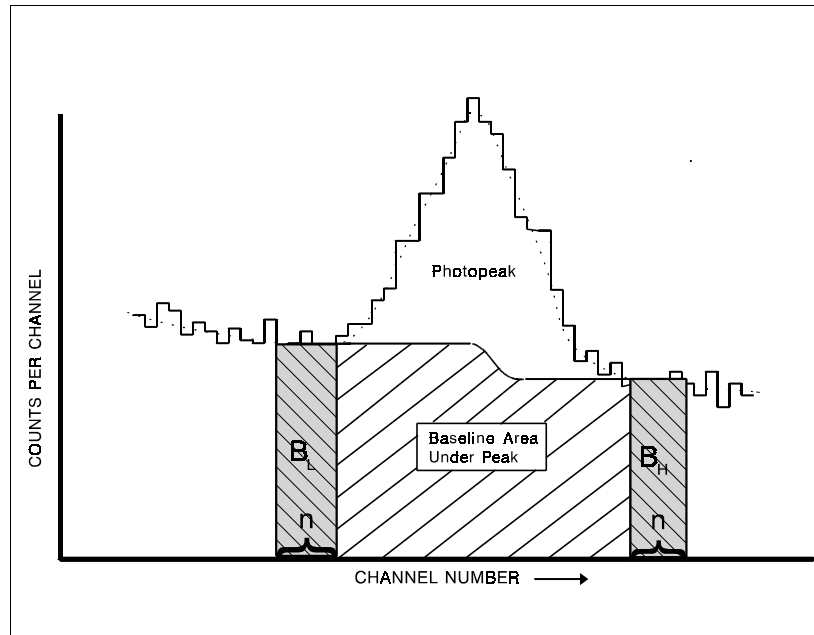


FIGURE 16.5 — Photopeak baseline continuum-step function

This type of function is estimated by:

$$B = \sum_{i=1}^N \left[\frac{B_L}{n} + \frac{B_H - B_L}{nG} \sum_{j=1}^i y_j \right] \quad (16.12)$$

where:

- B = the number of counts attributed to the baseline
- B_L = sum of counts in the baseline region on the low-energy side

- B_H = sum of counts in the baseline region on the high-energy side
- y_j = counts per channel in channel j
- G = gross counts in the peak
- N = number of channels in the peak
- n = number of channels in each of the two baseline regions

The second peak area determination method is the least-squares method, which fits a theoretical peak shape plus background shape to the channels surrounding the peak (Kruse and Spettel, 1982; Helmer and McCullough, 1983). Background is often subtracted prior to the fitting process (Loska and Ptasinski, 1994).

16.3.1.6 Calibration Reference File

Both energy and efficiency calibrations are required for gamma-spectrometric analysis. These calibrations require a source whose gamma-ray emission rate is known and traceable to a national standard, and whose gamma-ray energies are well-defined. “Mixed radionuclide” standards, containing eight or more gamma-ray energies (from a variety of radionuclides) are commercially available for performing these spectrometric calibrations. Information required for proper energy and efficiency calibrations include:

- Radionuclide;
- Activity at the analysis date and time (or a specified reference date);
- Analysis date and time;
- Half-life;
- Energy;
- Energy tolerance (energy window expressed as \pm keV);
- Gamma-ray emission per decay event (or “branching ratio”);
- Emission-rate uncertainty; and
- Desired activity units.

This information usually is included on the calibration source certificate provided by the manufacturer. Calibration files are created using the software and methods prescribed by the instrument manufacturer. One of the factors important to efficiency and energy calibrations that results from this process is the FWHM. This is a function of gamma-ray energy and is used in assessing peak shapes and areas in different regions of the energy spectrum. The values for FWHM from the gamma rays in the standard are also used to establish acceptable tolerance limits for gamma rays in the analyte corresponding to the energy regions of the calibration source.

16.3.1.7 Activity and Concentration

In order to convert the counts under a photopeak to activity, an efficiency calibration should be performed on the detector for each test-source geometry. Since the efficiency varies with energy, the detector should be calibrated over the range of energies to be used and a calibration curve developed for the detector. In constructing an efficiency calibration curve, only calibration sources with singlet peaks and well-known abundances should be selected. The efficiency, at a specific energy, is simply the number of counts determined in a photopeak of known energy divided by the number of gamma rays emitted by the source in the same time period, or:

$$\varepsilon = \frac{C_r}{D} \quad (16.13)$$

where:

- ε = efficiency (cps or γ ps)
- C_r = cps measured under the area of the photopeak
- D = gamma-ray emission rate of source (γ ps)

The efficiency versus energy curve developed in most gamma software packages is in the form of a polynomial. One such form is:

$$\ln \varepsilon = \sum_{i=0}^n b_i \cdot [\ln E]^i \quad (16.14)$$

where:

- ε = full peak efficiency
- n = degree of the polynomial
- b_i = coefficient as determined by calculation
- E = the energy of the photopeak

The efficiency curve for HPGe detectors is comprised of three distinct regions: a low-energy curve (up to about 120 keV), a middle-energy curve (from about 120 to 661 keV), and a high-energy curve (> 661 keV). Frequently, manufacturer-installed software can be used to generate a single continuous efficiency curve from the three separate regions.

This efficiency curve is maintained in the calibration file of the spectral analysis program to be applied to each analysis. An efficiency curve should be maintained for each test-source geometry to be used for the calibrated detector.

To obtain the activity in the test source, the net counts (background subtracted) in the photopeak, as determined by the software through the process described above, is divided by the geometry-specific efficiency. The activity units are converted to those selected by the operator and corrected for decay to the time of collection. Based on sample-aliquant size/volume information

supplied by the operator, sample concentration is calculated and reported.

16.3.1.8 Summing Considerations

Summing refers to the summing of the energy of two or more gamma rays when they interact with the detector within the resolving time of the spectrometer's electronics. There are two types of summing: (1) Random summing, where two unrelated gamma rays are detected at the same time, and (2) true coincidence summing, which is due to the simultaneous emission of several gamma rays by a radionuclide and their subsequent detection by the gamma detector.

Random summing, sometimes referred to as "pile-up," is due to gamma-ray emissions from different atoms being detected almost simultaneously. If two gamma rays interact with the detector within the charge collection time of the detector or the resolving time of the amplifier, a count will occur in a single channel somewhere else in the spectrum equal to the sum of the two deposited energies. Random summing can occur for any pair of events, such as photoelectric with photoelectric, photoelectric with Compton, and Compton with Compton. Since this occurs randomly in nature, the probability of random summing increases with the square of the total count rate. Random summing can be reduced by the use of pile-up rejection circuitry, which examines the pulse shape of detector signals and rejects those that are distorted by summing (Gilmore and Hemingway, 1995). However, even with pile-up rejection random summing will still be present. A mathematical correction for random summing is given by:

$$A_T = A e^{2R\tau} \quad (16.15)$$

where:

- A_T = the true peak area (counts)
- A = the observed peak area (counts)
- R = the mean count rate of the total spectrum (cps)
- τ = the resolving time of the electronics (s)

If unknown, the resolving time can be estimated by a method similar to that described in Gilmore and Hemingway (1995).

True coincidence summing is a source of error when a source contains nuclides that emit several gamma rays nearly simultaneously. Coincidence summing is geometry dependent and increases as the source is positioned closer to the detector. Thus, the use of multi-gamma-ray calibration sources for close geometry efficiency calibrations must be done with caution. True coincidence summing also increases with detector volume and is very prevalent in a "well" detector. The use of a detector with a thin entry window opens the possibility of coincidence summing with X-rays. Since coincidence summing is independent of count rate, it is a mistake to assume that the measurement of environmental media is immune from errors caused by this phenomena.

True coincidence summing can result in the loss of counts from photopeaks that are in

coincidence, and an apparent loss in the number of events detected at those energies. The sum peak also may be an emitted gamma ray. In this case, it would appear that more counts are present than expected at the sum-peak energy based on other peaks from the same radionuclide. The use of single gamma-ray-emitting radionuclides is recommended, to the extent possible, for developing calibration curves for detectors at close geometries. In practice, even when the efficiencies are determined in this manner, errors in analyzing for nuclides emitting more than one gamma ray still exist. When a multi-emitting gamma-ray source is to be measured with minimum bias, it may be necessary to perform an efficiency calibration with the specific radionuclide to be measured in the specific geometry desired.

In theory it is possible to mathematically correct for true coincidence summing; however, for complicated decay schemes, the task is daunting (Gilmore and Hemingway, 1995). Some data have been published that give correction factors for coincidence summing for a number of radionuclides (Debertin and Helmer, 1988). Unfortunately they only apply to the particular detector and geometries for which they were developed.

16.3.1.9 Uncertainty Calculation

The various components of uncertainty in the determination of the source activity should be propagated to obtain the combined standard uncertainty. The sources of uncertainty in the gamma spectral analysis include those associated with the determination of the net peak area, which includes the standard uncertainties of the gross counts, the background counts, and any interference from other gamma radionuclides present; the uncertainty associated with the unfolding of multiplets; the detector efficiency, which includes uncertainties of the net peak area, the calibration source emission rate, and decay correction factor; and uncertainty in the determination of the sample volume or mass.

$$u_c = \sqrt{u_P^2 + u_V^2 + u_e^2 + u_U^2 + u_F^2 + u_D^2} \quad (16.16)$$

where:

- u_c = combined standard uncertainty
- u_P = component of combined standard uncertainty due to the net peak area determination
- u_V = uncertainty component for the volume or mass determination
- u_e = uncertainty component for the efficiency determination
- u_U = uncertainty component for the unfolding routine for multiplets
- u_F = uncertainty in the branching factor from the decay scheme for the radioactive emission being measured
- u_D = uncertainty in the decay constant of the radionuclide

Each of these factors may have a number of components of uncertainties included, for example, the net peak uncertainty:

$$u_P = \sqrt{u_G^2 + u_B^2 + u_E^2 + u_I^2} \quad (16.17)$$

where:

- u_G = the uncertainty component for the gross counts in the peak
- u_B = the uncertainty component for the baseline subtraction
- u_E = the uncertainty component for the background peak subtraction
- u_I = the uncertainty component for the coincidence summing correction

The calculations of combined standard uncertainty typically are performed by the gamma-ray spectrometry software. It should be noted that not every available software package will incorporate all the listed uncertainty contributions listed.

16.3.2 Alpha Spectrometry

This section deals with alpha-spectrum reduction as applied to semiconductor detectors. The range of alpha particles in air is only a few centimeters, and their energy degrades significantly only after a few millimeters. Therefore, alpha spectrometry is conducted in a partial vacuum and on extremely thin sources prepared by electrodeposition or coprecipitation. Typically, an alpha spectrometry system is set up to generate spectra from such thin sources that cover an alpha energy range between 4 and 10 MeV (see Chapter 15).

The number of full energy peaks is usually not large, three to four, in an alpha spectrum, and they are normally well separated in energy. This, coupled with the fact that the test source subjected to counting has gone through a chemical separation (Chapter 14), makes the radionuclide identification relatively simple when compared to gamma-ray spectrometry. However, it is still of great benefit to have alpha spectrometry software to identify radionuclides, subtract background, perform calibrations and energy alignments, determine radiochemical yields, and perform and track QC functions. In production laboratories where hundreds of alpha spectra may be generated each week, it is almost imperative that alpha spectra are resolved by properly designed computer software. An alpha spectrum produced by a semiconductor detector by the counting of a thin source containing ^{234}U , ^{238}U , ^{239}Pu , and ^{241}Am is shown in Figure 16.6.

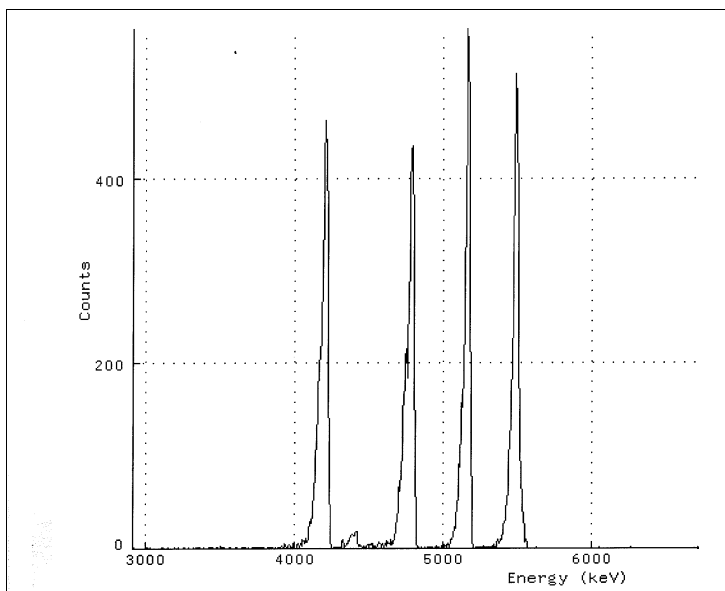


FIGURE 16.6 — Alpha spectrum
 $(^{238}\text{U}, ^{235}\text{U}, ^{234}\text{U}, ^{239/240}\text{Pu}, ^{241}\text{Am})$

The shape of each of the five peaks in the figure appears superficially Gaussian but actually differs from the pure Gaussian model for a number of reasons. One reason is that each of the alpha-emitting radionuclides emits alpha particles at more than one energy. So, each apparent peak is actually a combination of several peaks, whose energies are too close together to be resolved by the spectrometer. A second reason is that each peak has a low-energy tail caused by degradation of the energies of alpha particles as they pass through matter. Very thin, flat, nearly massless sources tend to produce the smallest tails. A third reason is that some peaks also have noticeable high-energy tails, which can be caused by the summing of alpha-particle energies with the energies of conversion electrons associated with the alpha decay. Note that the baseline for all the alpha peaks is essentially zero. An alpha-particle spectrum differs from a gamma-ray spectrum in that it does not have a background component comparable to the Compton continuum.

Spectral analysis programs usually have routines to identify full-energy peaks. In the case of alpha spectrometry, because the number of alpha peaks is limited and their energies are well known, a simple ROI-type of analysis usually is performed. Peak-fitting programs are available and may be beneficial when peak overlap is of concern. The alpha-peak deconvolution algorithms should take into account the low-energy tailing (Equation 16.9). The algorithms that account for tailing are modified Gaussian functions and require a peak-shape calibration where a number of well-defined singlet peaks covering the full energy range are acquired. The calibration program then calculates the tail-parameter values (see the discussion on tailing in Section 16.3.1.4, "Peak Width Determination"). These programs should be applied with caution to spectra where the peak tails are misshapen or non-normal. The uncertainty due to the fitting algorithm can create unexpected results. The goodness-of-fit at the top of the alpha peak and at the low-energy tail should be reviewed carefully before accepting their results. If the algorithm is not providing reasonable results, the analyst may choose to seek alternatives to these algorithms to improve spectral resolution. These may include counting the sample at a distance farther from the detector or performing additional chemical separations to improve radiochemical purity.

Alpha peaks are normally sitting on the baseline (no background continuum) and display minimal overlapping for well-prepared sources. For a given analysis (Pu, U, Am, Th, and etc.), ROIs are established for all energies of the alpha emissions in the source being counted and the count rate in a given ROI represents the emission rate of the alpha whose energy falls within that ROI. However, it is important to establish QC limits for the alpha resolution parameter for the test sources being analyzed. For example, the alpha resolution should be held to less than 90–100 keV FWHM in order to prevent significant overlapping between the ^{243}Am tracer (11 percent at 5.233 MeV and 88 percent at 5.274 MeV) and the ^{241}Am peaks (13 percent at 5.443 MeV and 85 percent at 5.486 MeV). The test source typically is counted to achieve at least 1,000 counts (3 percent uncertainty) in the ^{243}Am tracer peak, which should be sufficient to estimate the alpha resolution. A laboratory may remount (microprecipitation method) or replate (electroplate method) the test source if the resolution exceeds an established QC limit.

Given these qualifications, the spectral analysis software performs essentially the same functions as for gamma analysis, described above. The programs may also perform system control function, e.g., maintaining vacuum in the chambers. Databases related to procedures, chemical tracers, and efficiency and energy calibration standards are normally maintained for calculation, documentation, and QC purposes. The general analysis sequence for alpha spectrometry is discussed briefly below.

If a standard reference material is used for a tracer in each sample and an accurate determination of the yield is not required, an efficiency calibration is not necessary. In some cases, the laboratory may perform an energy and efficiency calibration for an alpha spectrometry analysis. This requires the operator to establish a calibration certificate file for the program to reference. It should refer to this file for both energy and efficiency calibrations. Calibration sources are necessary for performing the required calibrations, and the appropriate certificate information should be entered into the certificate files in order to perform the calibrations and to analyze test sources. This information should be supplied with calibration sources. Calibration sources, consisting of three to four radionuclides, are available in the form of plated discs from several commercial suppliers.

Information typically required by the analysis program consists of the following:

- Radionuclide
- Activity at the analysis date and time (or a specified reference date)
- Analysis date and time
- Half-life
- Energy
- Energy tolerance (energy window expressed as \pm keV)
- Alpha-particle emission per decay event (or “branching ratio”)
- Emission rate uncertainty
- Activity units desired

This information should be entered for each of the radionuclides included in the calibration source. Once the library file has been established, an energy calibration can be performed as directed by the software program. Some projects may require the reporting of the detector efficiency and chemical yield separately for each sample. For such cases, a one-time, initial calibration typically is determined and reported for each detector in use. When a calibration source contains several radionuclides with certified activities, a weighted mean efficiency should be calculated for the full-energy peaks and used as the alpha efficiency for a given detector (Chapter 15). The weighting factor would be the inverse of the variance (one over the square of the combined standard uncertainty) in the calculated detector efficiency for a radionuclide (Chapter 19).

The efficiency for alpha particles varies only slightly with energy, within the range of alpha

energies usually encountered (4-10 MeV). While the calibration source may contain several certified radionuclides, during an efficiency calibration, the mean efficiency for the full-energy peaks may be calculated and used as the alpha efficiency for a given detector (Chapter 15).

Once the alpha spectrometry system has been calibrated and a spectrum of a test source acquired, either a peak search is performed to identify alpha peaks or, if operating in a ROI mode, the counts in the ROI are determined. ROIs to be used for a given analysis are established prior to the spectrum acquisition by selecting an analysis protocol where the radionuclides and their alpha energies are preestablished.

In the ROI mode, the counts accumulated during the preset counting duration in each of the designated regions are corrected for background contribution and, in some cases, for reagent blank activity. If a tracer has been added to the test source, the counts in the tracer ROI are summed, background-corrected, and the effective efficiency (yield times counting efficiency) determined using certificate information previously entered by the operator or from a protocol file. The yield, if required, is then computed by the use of an efficiency that has been determined previously during an efficiency calibration process. The radionuclide concentration is then calculated by³:

$$R_{C_i} = \frac{C_{R_i}}{\epsilon_e \cdot V \cdot e^{-\lambda_i t_1}} \quad (16.18)$$

where:

- R_{C_i} = radionuclide concentration of the radionuclide at time of collection (Bq/L or Bq/g)
- C_{R_i} = net count rate in the designated ROI for the radionuclide (cps)
- ϵ_e = effective efficiency ($\epsilon \cdot Y$) for the tracer (cps or Bq)
- V = volume or mass analyzed (L or g)
- e = base of natural logarithm
- λ_i = radioactive decay constant for the radionuclide (reciprocal time units)
- t_1 = time lapse from sample collection to beginning of source count (units consistent with λ_i)

Following the spectrum acquisition process, spectral analysis programs may either automatically process the data and present the results, or they may store the spectral data and await interaction from the operator for processing. In either case, post-acquisition review of the analysis results is recommended. This review may include the following items:

³ For short-lived alpha-emitting radionuclides (e.g., ²²⁴Ra), a correction factor is needed for decay during counting. See Attachment 14A, "Radioactive Decay and Equilibrium."

- Assuring that the alpha peaks fall within the ROIs;
- Confirming the absence of unexpected peaks (contamination);
- Verifying that there are no interfering peaks;
- Confirming that peak centroids are within requirements (energy alignment);
- Verifying that all requirements are met with regard to FWHM (if possible) and chemical yield; and
- Checking units and sample aliquant information.

The FWHM of a given peak may depend greatly on the source preparation. However, since an ROI-type of peak search is normally used, and the limits of the peak determined by the setting of the ROI rather than some algorithm, the peak width definition is not significantly affected by reasonable peak broadening. As a precautionary measure, the above review of each test-source spectrum assures that the peaks appear within the ROIs. Alpha spectrometry analysis software allows for the adjustment of the ROIs to account for peak broadening and slight displacement. A review of the FWHM of the alpha peaks, as calculated by the software, will also reveal peak broadening due to matrix effects and poor test-source preparation.

16.3.2.1 Radiochemical Yield

Alpha spectrometry test sources are usually prepared by radiochemical separation and the chemical yield may be less than 100 percent. Therefore, a radiochemical tracer, which is an isotope of the radioactive species for which the analysis is being performed, may be added to the sample prior to preparation and radioanalysis. The tracer is normally a certified standard solution whose recovered activity is determined during the alpha spectrometric analysis in the same manner as the activities of the isotopes for which the analysis is being performed. The radiochemical yield is then calculated by the spectral analysis program according to:

$$Y = \frac{A_R}{A_S} \quad (16.19)$$

where:

Y = radiochemical yield

A_R = calculated activity recovered

A_S = certified activity added (decay corrected to time of counting)

The calculation of the chemical yield is normally performed by the alpha spectrometry analysis software using operator input information relative to the alpha energy and abundance, activity, uncertainty, and date of certification of the radiochemical tracer.

For some types of radionuclide analyses, no suitable alpha-emitting radionuclide may be available for use as a chemical yield tracer. In this case, the chemical yield may be determined by some other method, such as beta counting, and the resulting yield value provided to the alpha

analysis program so the source activity may be calculated from the alpha spectrometry data.

When a certified reference material is used for the chemical tracer, the effective efficiency is measured for each test source. If the chemical yield is to be reported, an independent measure of the counting efficiency should be made.

16.3.2.2 Uncertainty Calculation

The calculation of the combined standard uncertainty for alpha spectrometry is similar to that for gamma-ray spectrometry as reported in Section 16.3.1.8 above. One additional source of uncertainty that should be taken into account for alpha spectrometry is that associated with the determination of radiochemical yield. Since a tracer is added to the sample and the yield determined by a counting process, the uncertainty involved in this analysis should be accounted for in the total uncertainty. The uncertainty of the yield determination involves that associated with the net count of the tracer, the counting efficiency, and that of the emission rate of the tracer material. The combined standard uncertainty of the radionuclide concentration, R_{C_i} , is given by either

$$u_c(R_{C_i}) = \sqrt{\frac{u^2(C_{R_i})}{\epsilon_e^2 V^2 e^{-2\lambda_i t_1}} + R_{C_i}^2 \left(\frac{u^2(V)}{V^2} + \frac{u^2(\epsilon_e)}{\epsilon_e^2} \right)} \quad (16.20)$$

or

$$u_c(R_{C_i}) = \sqrt{\frac{u^2(C_{R_i})}{\epsilon^2 Y^2 V^2 e^{-2\lambda_i t_1}} + R_{C_i}^2 \left(\frac{u^2(V)}{V^2} + \frac{u^2(\epsilon)}{\epsilon^2} + \frac{u^2(Y)}{Y^2} + \frac{2u(\epsilon, Y)}{\epsilon \cdot Y} \right)} \quad (16.21)$$

where:

- C_{R_i} = net count rate in the designated ROI for the radionuclide (cps)
- ϵ = the alpha counting efficiency
- Y = the chemical yield
- ϵ_e = effective efficiency ($\epsilon \cdot Y$) for the tracer (cps or Bq)
- V = volume or mass analyzed (L or g)
- e = base of natural logarithm
- λ_i = the radioactive decay constant for the radionuclide (reciprocal time units)
- t_1 = time lapse from sample collection to beginning of source count (units consistent with λ_i)

$u(\cdot)$ denotes the standard uncertainty of a quantity

$u(\cdot, \cdot)$ denotes the covariance of two quantities

The two uncertainty equations are equivalent. However, when the yield is determined using an alpha-emitting tracer, Equation 16.20 generally is easier to implement.

16.3.3 Liquid Scintillation Spectrometry

16.3.3.1 Overview of Liquid Scintillation Counting

All modern counters are computer controlled for data acquisition, spectral unfolding, data reduction, sample changer control, external quench correction, and performing the various other functions associated with liquid scintillation counting.

Liquid scintillation has traditionally found its primary use in the analysis of low-energy beta emitters, such as ^3H and ^{14}C . In spite of the complicating factors of high background and quenching (Section 15.4.5.4), procedures for other beta- and alpha-emitting radionuclides have been developed over the years (Holm et al., 1984; Harvey and Sutton, 1970).

Liquid scintillation has also been applied to the simultaneous analysis of alpha and beta emitters in environmental media (Leyba, 1992). Discrimination between alpha and beta radiation is based on differences in the fluorescence decay pulses. Pulse height is proportional to particle energy, and high counting efficiency results from 4π (4-pi) geometry and the absence of test-source self-attenuation (McDowell and McDowell, 1993). Because of these characteristics, liquid scintillation counting can be utilized as an alternative to proportional counting (Section 16.4) and alpha semiconductor counting (Section 16.3.2).

16.3.3.2 Liquid Scintillation Spectra

The amount of light produced by alpha and beta particles in a liquid scintillation cocktail is proportional to the particle energy. Beta spectra convey the energy continuum from zero to their maximum energy. Alpha liquid scintillation spectra are similar in shape to those obtained by semiconductor spectroscopy, but with greatly decreased resolution. Because alpha particles are only about one-tenth as efficient as beta particles in producing scintillation light pulses, there is an overlap of alpha and beta spectra (Passo and Kessler, 1992; McDowell and McDowell, 1993).

Gamma radiation interactions within the scintillation cocktail depend on energy and path length, with lower energy gamma rays being more efficient in transferring their energy. Gamma events are recorded in the same energy range as alpha and beta particles; therefore, discrimination between alpha, beta, and gamma radiation based solely on scintillation spectra is not possible (Passo and Kessler 1992; McDowell and McDowell, 1993).

16.3.3.3 Pulse Characteristics

Excited triplet and singlet energy states are formed by the fluor molecules when ionizing radiation interacts with the scintillation cocktail. The excited singlet states dissipate their energy very rapidly and produce short lifetime decay pulses, whereas triplet states lose their energy more slowly, resulting in longer lifetime pulses. Because alpha particles have a higher linear energy

transfer than gamma or beta radiation, they produce a higher ratio of triplet to singlet excitation states and therefore have a longer pulse duration. Differences in the decay time and shape of the decay pulse are the basis for discriminating alpha particles from beta and gamma radiation in liquid scintillation counting (Passo and Kessler 1992; Passo and Cook 1994).

16.3.3.4 Coincidence Circuitry

Most modern liquid scintillation counters employ two photomultiplier tubes 180 degrees apart for the detection of pulses. The light produced when ionizing radiation in the test source interacts with the scintillation cocktail is emitted in all directions. A sample event should therefore produce electronic pulses in both photomultiplier tubes simultaneously, or in coincidence.

Electronic noise pulses are produced randomly by the photomultiplier tubes, but the probability that both tubes will produce noise pulses simultaneously is very low. An electronic gate can be set to allow only pulses that are in coincidence to be registered. The rejection of random pulses keeps background counts produced by electronic noise to a minimum. Similarly, the probability of background radiation (such as cosmic radiation) yielding an event in both photomultiplier tubes is remote due to the coincidence circuitry.

16.3.3.5 Quenching

Quenching is discussed in detail in Section 15.4.5.4. Chemical quenching reduces the amount of energy transferred to the fluor molecules. Halogens, water, solvents, some acids, and oxygen are common agents that cause a decrease in the counting efficiency.

Color quenching is caused by impurities not removed during test-source preparation or by carrier compounds such as iron chloride. Photons emitted from the fluor molecules are absorbed, reducing the amount of light reaching the photomultiplier tubes.

Quenching causes a shift in the scintillation spectrum to lower energies and a reduction in the number of counts. Quenching has a minimal impact on alpha counting, but significantly increases as the energy of the beta particle decreases.

The most common method for monitoring sample quench is through the analysis of a Compton spectrum generated by gamma rays interacting with the sample-scintillation cocktail. After the test source is loaded into the counter, it is irradiated by an external gamma emitting source located in the instrument. The test-source spectrum is collected and compared with factory or user-generated quench standards stored in the instrument library. Both color and chemical quenching cause a shift to lower energies, but the color quench broadens the spectrum as well. The efficiency of the test source is extrapolated and applied to normalize the test-source count rate.

16.3.3.6 Luminescence

Photoluminescence is produced by ultraviolet light from the environment reacting with the scintillation cocktail. The effect can be minimized by dark adapting the test sources prior to counting.

Chemiluminescence is produced by reactions between the scintillation cocktail and chemicals introduced from the test-source preparation. To minimize this effect, oxidizers and alkaline conditions should be avoided.

Both photoluminescence and chemiluminescence cause random scintillation events. At low levels, the coincidence gate should reject most of their contribution. However, at very high levels, the probability increases that two events may pass through the gate. Manufacturers use a method of spectral stripping to correct for the false counts, but it is best to avoid the conditions that create the problem.

16.3.3.7 Test-Source Vials

Glass test-source vials contain naturally occurring impurities such as ^{40}K , Th, and U. Their contribution appears at the lower energy portion of the spectrum. Plastic vials have a lower background, but they should be compatible with the liquid scintillation cocktail being used. Teflon[™] vials are also available from most manufacturers.

16.3.3.8 Data Reduction for Liquid Scintillation Counting

Liquid scintillation counters normally provide minimal data reduction in their output. Basic data include the counting duration, count rate in one or more selected windows, and the date and time of counting initiation. A blank source (background), having a similar quench factor as the test sources, normally is counted with each counting batch and the output will provide the count rate of the blank to be subtracted from each test source.

The detecting efficiency will also be provided by the output information. Its form of presentation in the output will depend on the calibration/counting (quench correction) method for determining detector efficiency⁴. If the internal (standards addition) method is used, the data generated by the counter must be further manipulated in order to develop the counting efficiencies for each test source. When using the external-standards method (quench curve), the scintillation spectrometer will apply the quench corrected efficiency and give the test sample disintegration rate by applying the corrected efficiency.

⁴ For a discussion of liquid scintillation efficiency determination, see Section 15.5.3.

The radionuclide or gross concentration is provided by the following equation:

$$A_C = \frac{C_G - C_B}{\epsilon_q V} \quad (16.22)$$

where:

- C_G = gross counting rate (source + background) (cps)
- C_B = counting rate of the blank (cps)
- ϵ_q = radionuclide quench-corrected counting efficiency for the specific radionuclide (cps or Bq)
- A_C = radionuclide or gross concentration (Bq/L or Bq/g)
- V = volume or mass analyzed (L or g)

16.4 Data Reduction on Non-Spectrometry Systems

Proportional counters are primarily used for counting of test sources for alpha and beta emitters. Proportional counters may have entry windows for allowance of the emitted radiation into the active portion of the detector or they may be windowless. These instruments are described in Chapter 15. They are used for the determination of specific radionuclides, following chemical separation to isolate the radionuclide, and for nonspecific (gross) analyses. Counters are equipped to count alpha and beta simultaneously in a given source and report the activity of both.

The basic information obtained from a determination in a proportional counter is the number of counts recorded in the detector within the allotted counting duration. However, modern proportional counters take the data reduction process to the point of finality, i.e., producing the test-source concentration and associated counting uncertainty, providing automatic instrument background subtraction, and correcting for source self-absorption and alpha/beta crosstalk.

The instruments may also have protocols for developing the correction factors for self-absorption and for crosstalk. In addition, they should have the capacity to track and evaluate the periodic QC checks (check source and background) performed on the instrument.

The basic equation used to calculate test-source activity is:

$$A = \frac{C_G - C_B}{\epsilon} \quad (16.23)$$

where:

- A = the activity of the radionuclide or gross activity (Bq)
- C_G = the gross counting rate (source + background) (cps)

- C_B = the instrument background counting rate (cps)
- ϵ = the gross or radionuclide counting efficiency (cps or Bq)

And the radionuclide or gross concentration is provided by the following equation:

$$A_C = \frac{C_G - C_B}{\epsilon V} \quad (16.24)$$

where:

- A_C = radionuclide or gross concentration (Bq/L or Bq/g)
- V = the volume or mass analyzed (L or g)

The associated combined standard uncertainty is given by:

$$u_c(A_C) = \sqrt{\frac{u^2(C_G) + u^2(C_B)}{\epsilon^2 V^2} + A_C^2 \left(\frac{u^2(\epsilon)}{\epsilon^2} + \frac{u^2(V)}{V^2} \right)} \quad (16.25)$$

The above simple equations apply to counting either pure alpha or beta emitters and when no correction for self-absorption is necessary (weightless sources). Modifications should be made in the activity and concentration calculations when both alpha and beta particles are emitted by the source, and when absorption and scattering within the source cause a reduction in the effective efficiency.

Self-absorption factors are applied for sources where the self-absorption of the alpha or beta particle is sufficient to affect the overall efficiency (Chapter 15). Commercially available proportional counters have a protocol for developing the self-absorption correction factors. These protocols process the data generated by counting a series of alpha calibration sources and a series of beta calibration sources, which both have varying masses of material, from “zero” to the maximum to be encountered in test sources (Chapter 15). The instrument is programmed to then fit the data to a mathematical function so the counting efficiency correction factor can be applied at any test-source mass within the range covered by the calibration source masses. A cubic polynomial is one option used for both alpha and beta counting efficiencies. A cubic polynomial has the form

$$\epsilon_m = a_0 + a_1 m + a_2 m^2 + a_3 m^3 \quad (16.26)$$

where:

- m = is the residual mass of the test source
- ϵ_m = the counting efficiency at mass m
- a_i = constants determined by the data fit

The combined standard uncertainty of ϵ_m is given by

$$u_c(\epsilon_m) = \sqrt{u^2(a_0) + \sum_{i=1}^3 m^{2i} u^2(a_i) + 2 \sum_{i=0}^2 \sum_{j=i+1}^3 m^{i+j} u(a_i, a_j) + (a_1 + 2a_2 m + 3a_3 m^2)^2 u^2(m)} \quad (16.27)$$

When the identities of the alpha or beta emitting radionuclides are unknown, an additional component of uncertainty is needed to account for the dependence of the counting efficiency (and self-absorption) on the unknown particle energy.

Another option that is often used for the beta counting efficiency is an exponential curve, which has the form

$$\epsilon_m = \epsilon_{\text{zero}} e^{-am} \quad (16.28)$$

where:

- m = is the residual mass of the test source
- ϵ_m = the counting efficiency at mass m
- ϵ_{zero} = the “zero” mass counting efficiency
- a = constant determined by the data fit

Then the combined standard uncertainty of ϵ_m is:

$$u_c(\epsilon_m) = e^{-am} \sqrt{a^2 u^2(m) + u^2(\epsilon_{\text{zero}}) + m^2 u^2(a) - 2m u(\epsilon_{\text{zero}}, a)} \quad (16.29)$$

Again, an additional uncertainty component may be needed when the identity of the beta-emitting radionuclide is unknown.

Crosstalk, sometimes called “spill over,” refers to the misclassification of alpha- and beta-produced counts in a proportional counter that is designed to count both particles simultaneously. It occurs when counts produced by alpha interactions in the detector are registered as beta counts and vice versa. In order to accurately record the alpha and beta activities of sources containing radionuclides emitting both particles, corrections should be made for crosstalk.

The number of alpha interactions registered as beta counts will increase as the source self-absorption increases. The opposite is true for beta crosstalk, in that the number of beta interactions falsely designated as alpha counts decreases with source self-absorption. Thus, crosstalk correction factors vary with test-source mass and should be developed for the range of test-source masses to be encountered. Commercially available proportional counters have established programs to assist in the establishment of alpha and beta crosstalk factors. The algorithms to correct for crosstalk are presented below.

The alpha-in-beta crosstalk, $X_{\alpha\beta}$, is defined as:

$$X_{\alpha} = \frac{\beta}{\alpha + \beta} \quad (16.30)$$

The respective counts in the alpha channel (α) and those in the beta channel (β) counts are measured with a pure alpha-emitting source. Likewise, the beta-in-alpha crosstalk, X_{β} , is:

$$X_{\beta} = \frac{\alpha}{\alpha + \beta} \quad (16.31)$$

The respective alpha (α) and beta (β) count rates are measured with a pure beta-emitting source.

The relationship between X_{α} and X_{β} is given by:

$$\alpha = \alpha_d - \alpha_d X_{\alpha} + \beta_d X_{\beta} \quad (16.32)$$

$$\beta = \beta_d - \beta_d X_{\beta} + \alpha_d X_{\alpha} \quad (16.33)$$

Equation 16.32 states that the recorded alpha count rate, α , consists of the actual alpha count rate, α_d , (the total alpha count rate in both the alpha and beta channels due to only alpha interactions), minus those alpha interactions recorded in the beta channel, plus those beta counts recorded in the alpha channel. Equation 16.33 is the equivalent of Equation 16.32 for beta counts. Solving the equations simultaneously for α_d and β_d gives:

$$\alpha_d = \frac{\alpha - X_{\beta}(\alpha + \beta)}{1 - X_{\alpha} - X_{\beta}} \quad (16.34)$$

$$\beta_d = \frac{\beta - X_{\alpha}(\alpha + \beta)}{1 - X_{\alpha} - X_{\beta}} \quad (16.35)$$

Their associated combined standard uncertainties are:

$$u_c(\alpha_d) = \frac{\sqrt{u^2(X_{\alpha})\alpha_d^2 + u^2(X_{\beta})(\alpha_d - \alpha - \beta)^2 + u^2(\alpha)(1 - X_{\beta})^2 + u^2(\beta)X_{\beta}^2}}{1 - X_{\alpha} - X_{\beta}} \quad (16.36)$$

$$u_c(\beta_d) = \frac{\sqrt{u^2(X_{\beta})\beta_d^2 + u^2(X_{\alpha})(\beta_d - \alpha - \beta)^2 + u^2(\beta)(1 - X_{\alpha})^2 + u^2(\alpha)X_{\alpha}^2}}{1 - X_{\alpha} - X_{\beta}} \quad (16.37)$$

Since crosstalk factors vary with radionuclide, additional uncertainty components may be needed

when the identities of the alpha and beta emitting radionuclides are unknown.

Processors execute many other functions for instruments that do not perform spectrometry. These instruments include proportional counters, scintillation detectors, ionization chambers, and special instruments (Chapter 15). The functions performed by processors may include instrument control (sample change, gas flow control, etc.) and the calculations necessary to convert the basic counting information to final form data or to some intermediate step.

Data reduction functions that may be performed for scintillation detectors, ionization chambers, and special instruments include the following:

- Determining background and subtraction;
- Converting total counts to counts per second;
- Calculating activity using calibration data;
- Calculating concentration using activity and operator input data;
- Performing efficiency calibrations;
- Calculating counting and total uncertainty;
- Determining crosstalk and corrections;
- Determining self-absorption corrections;
- Determining radioactive decay corrections; and
- Performing QC functions (efficiency and background verification).

The output of manual systems usually requires further reduction to render it usable. The information generated by processor-based systems may also need further processing.

These additional calculations may be performed using a calculator or by a computer using general or custom software programs. The data may be electronically transferred to the processing computer by a local area network (LAN) or on a computer disk. In some cases the processing software may be part of the LIMS.

16.5 Internal Review of Data by Laboratory Personnel

The final review of analytical data by the laboratory should be according to the laboratory quality manual or other documented procedures to ensure that the data meets specified requirements. All final inspections and reviews of data should be performed, documented and archived. The review of analytical data may be performed on two levels: primary and secondary. Different people should perform these two levels. Primary and secondary reviewers should be designated by management.

16.5.1 Primary Review

Some elements to consider in the primary review are:

- Verifying that all tests requested were performed for all samples
 - Target radionuclides
 - Sample preservation
 - Required sample preparation (filtration of liquid samples, drying of soils, etc.)
- Comparing the actual sample (radiological) holding times to the holding times specified in the analytical methods; holding-time exceedences should be documented in the final report
- Verifying that the appropriate method was selected and performed
- Determining that the target radionuclides were correctly identified
- Verifying that data inputs for calculations were correct
 - Examining the calibration curve to determine that the criteria specified in the analytical method were met (or verifying radiotracer activity used)
 - Dates and times for reference/sample, analysis, ingrowth/decay, chemical separation
 - Sample volumes/mass, detector backgrounds or analytical blanks, radionuclide half life, etc.
- Checking for errors in transcription and data inputs for calculations (such as rounding procedures and correction factors)
- Checking, by independent hand calculations when possible and at a specified frequency, automated data results for correct quantification
- Reviewing measurements results for reasonableness
- Verifying that measurement results meet MQO requirements
- Examining QC sample results for acceptable performance
- Verifying that the analyst's notebook and/or project file—
 - accurately captures the final results
 - is readable
 - documents any deviations from the analytical method
 - contains the analyst's initials or signature (written or electronic) and the completion date of the analyses

16.5.2 Secondary Review

The secondary review includes many of the same considerations as the primary review; however, there are additional aspects that may be addressed:

- Verifying that a primary review has been conducted
- Verifying that the correct analytical method was performed
- Verifying that the correct software was used to calculate the measurement results
- Examining computerized printouts for completeness
- Examining the final results, including QC sample results to determine if all relevant data

have been included

- Preparing or reviewing the Case Narrative to be included in the data package
- Verifying that all required signatures are included

16.6 Reporting Results

Quality planning documents will give the level of data reporting required. This level will vary from simply reporting the analytical results to a complete reporting of all measurements, calibration data, documentation of the performance of laboratory processes, provision of certain instrument counting reports, and QC sample results and analysis. Another way of viewing this is as a tiered approach where preliminary studies or site surveys may only require a minimum of data reporting, while a final site survey may require a detailed reporting of the results. The necessary elements for data reporting are connected to the purpose for which the data will be used (data quality objectives).

MARLAP recommends that the reported value of a measurement result: (1) be reported directly as obtained, with appropriate units, even if it is are negative, (2) be expressed in an appropriate number of significant figures, and (3) include an unambiguous statement of the uncertainty. The appropriate number of significant figures is determined by the magnitude of uncertainty in the reported value. Each reported measurement result should include the value and an estimate of the combined standard uncertainty (ANSI N42.23) or the expanded uncertainty.

16.6.1 Sample and Analysis Method Identification

Sample data are normally reported by sample number, including both the field (project) and laboratory assigned identifiers. In addition, the submitting laboratory should be identified as well as the analysis method (ANSI N42.23). Other information that can assist in the review and interpretation of the data may be requested. This could include sample collection date (decay correction reference date), analysis date, chain-of-custody (COC) number, and site or project name.

16.6.2 Units and Radionuclide Identification

The individual radionuclides should be identified or, for gross analyses, the category, e.g., gross alpha/beta, should be reported. For gross alpha and beta measurements, it is common practice to cite the “reference” radionuclide used to calibrate the detector or generate the detector efficiency factors from self absorption curves (e.g., ^{137}Cs for gross beta analyses). In some cases, the reference radionuclide will be specified by the project manager. The citation to the reference radionuclide should be made on the analytical reports submitted by the laboratory.

Reporting units are likely specified by project planning documents. If not specified, when

possible, the International System of Units (SI) is preferred. However, since regulatory compliance levels are usually quoted in traditional radiation units, it may be appropriate to report in both SI and traditional units with one being placed within a parenthesis. Both the SI and non-SI units are shown in Table 16.1 for common matrices.

TABLE 16.1 — Units for data reporting

Matrix	In Non-SI Units	In SI Units	Conversion Factor From Non-SI to SI Units
Airborne Particulates and Gas	pCi m ⁻³	Bq m ⁻³	3.70×10 ⁻²
Liquids	pCi L ⁻¹	Bq L ⁻¹	3.70×10 ⁻²
Solids	pCi g ⁻¹	Bq g ⁻¹	37
Surfaces	dpm/100 cm ²	Bq/100 cm ²	1.67×10 ⁻²

16.6.3 Values, Uncertainty, and Significant Figures

The value, as measured, including zero and negative numbers, and the measurement uncertainty (either expanded uncertainty or the combined standard uncertainty) should be reported in the same units (Chapter 19). In general, environmental radiation measurements seldom warrant more than two or three significant figures for the reported value, and one or two significant figures for the uncertainty. MARLAP recommends that the measurement uncertainty be rounded to two significant figures, and that both the value and uncertainty be reported to the resulting number of decimal places (see Sections 19.3.7 and 19.3.9). For example, a value of 0.8961 pCi/L with an associated measurement uncertainty of 0.0234 should be reported as 0.896 ± 0.023 pCi/L. The minimum detectable concentration (MDC) should be reported to two significant figures (ISO, 1995; ANSI N42.23). It should be noted that rounding should only occur in reporting the final results.

16.7 Data Reporting Packages

Project planning documents (Chapter 4) and analytical statements of work (Chapter 7, *Evaluating Methods and Laboratories*) usually define the requirements of the final data submission. The reporting of laboratory data may vary according to laboratory, SOW, or data validation requirements. If the laboratory has been requested to report the results for several radionuclides (as many as 20 in some applications) for each sample ID, the results for the required radionuclides may be reported together on a single report form. (An exception to this is the reporting of results from gamma-ray analyses.) Many projects will specify a data package of reports and supporting information necessary to describe, document, and define the analytical process. Table 16.2 provides a fairly comprehensive list of elements that might be included in a radiochemical data package, although not all will be applicable to a single data package and some are only applicable during an audit.

TABLE 16.2 — Example elements of a radiochemistry data package

General Information	
<ul style="list-style-type: none"> • Table of contents (especially for large packages) • Laboratory name • Client name • Identification of project, SOW, etc. • Identification of the sample batch • A complete list of samples, including: <ul style="list-style-type: none"> – Client sample ID – Laboratory sample ID – Sample matrix – Collection date(s) – Date of receipt by laboratory • Verification of field sample preservation • Signed acknowledgment of data package completeness 	Supporting laboratory documentation, such as copies of: <ul style="list-style-type: none"> • Relevant logbook pages • Standard certificates • Bench sheets • Instrument printouts, spectrum graphs • Control charts and QC reports (including instrument QC)
Batch-Specific Information	
<ul style="list-style-type: none"> • Unambiguous identification of the sample preparation and analytical procedures • Narrative describing how samples were received and processed • Notes of problems encountered (e.g., shipping problems, QC failures, deviations from the SOW or SOPs) • Explanations of terms, acronyms, other aspects of the report that may be unclear to the client • Identification of all preparation batches • Identification of analyst(s), either by batch or by sample • QC linkages (which QC samples go with which samples) 	
Analysis-Specific Information (For each test performed on an actual sample or QC sample)	
<ul style="list-style-type: none"> • Laboratory sample ID • Preparation batch ID • Size of sample aliquant • Which portion or fraction of sample was tested (if applicable; e.g., filtrate, undissolved solids) • Chemical yield (if appropriate), with uncertainty 	
For Each Instrumental Analysis	For Each Analyte Measured
<ul style="list-style-type: none"> • Instrument ID • Type and description • Date of most recent calibration • Duration of analysis • Description of test-source geometry 	<ul style="list-style-type: none"> • Analyte identifier • Measured result • Measurement unit • Uncertainty (with coverage factor) • Critical value • Minimum detectable concentration • Reference dates and times (for decay and ingrowth corrections)
QC-Sample Information	Chain-of-Custody Information
<ul style="list-style-type: none"> • Type of QC sample (matrix spike, etc.) • Numerical value of performance indicator • Pass-or-fail evaluation 	<ul style="list-style-type: none"> • Chain-of-custody or other tracking documents provided with samples or generated by the laboratory identifying the dates and times of the sample history

16.8 Electronic Data Deliverables

Many project planning documents and SOWs require that laboratory data be delivered in electronic format, commonly called electronic data deliverables (EDD). This allows the data to be transferred directly into a project database or, in some cases, into validation/review programs, and avoids transcription errors. Many of the elements in Table 16.2 may be reported electronically, but the record structure may vary in terms of database compatibility, field length, field order, and field name. Because there is no universal structure for EDDs, the laboratory may be required to produce them in various formats.

EDDs may be transmitted by direct electronic transfer, e-mail, or on removable media (disks, tapes, cartridges, CD-ROMs, removable hard drives). More information may be found at the following websites:

- The U.S. Department of Energy's Environmental Management Electronic Data Deliverable (EMEDD) may be found at ersmo.inel.gov/edd/.
- The U.S. Environmental Protection Agency's Environmental Data Registry is available at www.epa.gov/edr/.
- The U.S. Air Force Environmental Resources Program Management System (ERPRIMS) website (www.afcee.brooks.af.mil/ms/msc_irp.asp) also provides useful information on environmental databases and EDDs.

One EDD that promises to become a widely adopted format is the Staged Electronic Data Deliverable (SEDD) being developed jointly by the U.S. Army Corps of Engineers and the U.S. Environmental Protection Agency. SEDD is based on XML (eXtensible Mark-up Language) technology, which is a World Wide Web Consortium (www.w3.org) standard. SEDD will be adaptable for use with any agency or program database format through the use of parsers that transfer data from SEDD to the appropriate database elements. With the widespread adoption of SEDD, laboratories should need to produce only one type of EDD. Information on SEDD can be found at www.epa.gov/superfund/programs/clp/sedd.htm.

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