16 INSTRUMENT CALIBRATION AND TEST SOURCE PREPARATION

16.1 Introduction

This chapter provides guidance on the important functions of radiation detection instrument calibration and test source preparation. In this chapter, the term "test source" will be used to describe the radioactive material prepared to be introduced into a measurement instrument, and "laboratory sample" will be used to identify the material collected for analysis. Thus, a test source is prepared from laboratory sample material for the purpose of determining its radioactive constituents. "Calibration source" is used to indicate that the prepared source is for the purpose of calibrating instruments.

The continuing validity of calibrations should be checked on a periodic basis (Chapter 18, *Laboratory Quality Control*) as specified in a laboratory's quality assurance manual. This is usually done by counting a check source or some secondary standard in an instrument and comparing the results to those previously obtained when the instrument was known to be in calibration. The frequency and other aspects of calibrations and verifications may be specified in project planning documents (Chapter 4, *Project Plan Documents*) and in analytical statements of work (Chapter 5, *Obtaining Laboratory Services*).

Test sources may be prepared by destructive or nondestructive techniques. A destructive analysis is performed when the original laboratory sample material is altered by ashing or dissolution, which often is followed by chemical separations. Chemical separation usually is necessary when analyzing for specific alpha- or beta-particle emitters. Nondestructive analyses can be used when the laboratory sample is to be analyzed by gamma spectrometry or for gross analyses where the laboratory sample is only dried and counted directly.

The requirements placed upon test source preparation are dictated primarily by the type and energy of the radioactivity to be measured (alpha, beta, or gamma), the radiation detector employed, and—to some degree—whether the measurement is simply a gross radioactivity measurement or if specific radionuclide identification is required. The nature of the laboratory sample material also will have an effect on the test source preparation. These are referred to as "matrix effects" and can be caused by both the chemical and physical characteristics of the laboratory sample. When matrix effects are encountered, one is faced with the choice of altering the analysis methodology for that laboratory sample or possibly flagging the result to indicate a high degree of uncertainty.

The significant characteristics affecting the bias and precision of radioactivity measurements will be discussed in relation to each type of radioactivity. This includes counting efficiency, which can be affected by the characteristics of the test source as well as those of the radiation detector and the geometry of the source relative to the detector. Also, methods used to prepare radioactive test sources for measurement from chemically separated (isolated) radionuclides will be described.

A number of methods and techniques employed to separate and purify radionuclides contained in laboratory samples, particularly in environmental samples, are described in Chapter 14 (*Separation Techniques*), and sample dissolution is discussed in Chapter 13 (*Sample Dissolution*). Instruments that will be used to analyze the test sources prepared as outlined in this chapter are described in Chapter 15 (*Nuclear Counting Instrumentation*). In the case of gross (non-nuclide specific) and nondestructive measurements, chemical separation and purification procedures often are not required. However, to accomplish these measurements, the test source still must be prepared (mounted) in such a manner that the associated radioactivity can be quantified in a reproducible and unbiased manner.

16.2 Instrument Calibration

Instrument calibrations generally are performed for the purpose of establishing the counting efficiency of an instrument. The counting efficiency establishes the number of disintegrations registered in the detector and electronics of a counting instrument compared to the number emitted by the source. Counting efficiencies are specific to the radionuclide (or energy), the geometrical relationship between the source and detector, and a number of characteristics of the source material, especially those that affect absorption and scattering of the radiation. It is common practice to have several different calibrations on a given detector in order to accommodate a number of radionuclides, source-to-detector distances, and counting containers that a laboratory will be required to employ in order to meet project requirements for detection sensitivity, specificity, and the variety of media encountered.

In cases where the efficiency of the detector varies with energy, it is necessary to perform the calibration at a number of energies and establish an efficiency curve that covers the range of energies to be encountered. Some radiation detection instruments require other types of calibrations. These will be discussed under specific instrument calibrations. Generic issues which govern the conduct of calibrations will be discussed below and instrument and test source specific considerations will be provided in the appropriate sections in this chapter.

16.2.1 Standards

Instrument calibration should be performed as needed with only National Institute of Science and Technology (NIST) traceable standards (ANSI N42.23). Calibrations of instruments shall be made using certified reference materials of known and documented value and stated uncertainty. These certified reference materials shall be supplied by:

- NIST directly;
- A standard source supplier whose measurement capabilities or manufacturing processes are periodically tested by NIST; or
- A standard source supplier who documents derived materials with stated uncertainty, and whose value has been verified with analytical and measurement systems that have been tested periodically through an unbroken chain of comparisons to the national standards.

16.2.2 Correspondence

To assure that the instrument calibration is unbiased, calibration sources must be prepared and counted in a manner that assures that they are virtually identical to the test sources in all respects that could affect the counting efficiency determination (ANSI N42.23). The geometry, including the size and shape of the calibration source and counting container (beaker, planchet, vial, etc.) and source-to-detector distance and alignment, must be controlled. Backscatter, scattering, and self-absorption present during test source counting must be duplicated in the calibration process. The density of the calibration source material should be consistent with that of the test sources.

When possible, counting efficiency calibrations should be performed using the radionuclide, whose activity is to be determined in test sources. This may not be possible when the radionuclide is not available as a standard reference material or when gross analyses are performed. When the actual radionuclide is not available, a surrogate radionuclide may be selected that has the same type of particle or photon emission (α, β, or γ) and a proximate energy. When calibrating an instrument in this manner, corrections must be made for any differences between the decay schemes of the two nuclides.

If any factor can vary throughout the test sources, calibrations must be performed which simulate this variability over the range expected to be encountered during test source counting. An example is the necessity to develop a self-absorption curve for alpha or beta counting to account for the changing overall counting efficiency due to absorption in the variable source thickness.

16.2.3 Homogeneity

The calibration source must be prepared in a manner that assures that the material is uniformly distributed throughout its volume. Any deviation from this requirement can result in a calibration that is biased and contributes to the overall uncertainty of the laboratory results.

Liquid calibration sources are more likely to be homogeneous than are solids, particularly those where reference material has been added to a solid material—soil, for example. In order to minimize the overall uncertainty associated with calibration, care should be taken to assure the reference material is thoroughly mixed into the calibration source and distributed uniformly throughout its volume.

16.2.4 Uncertainty

The total uncertainty of calibration is affected directly by the uncertainty associated with the activity of the reference material used in the calibration source. Furthermore, the uncertainties related to the reproducibility of the counting geometry and the non-homogeneity of the calibration source must be considered. Since the uncertainty associated with these factors is difficult to quantify, it should be minimized.

The uncertainty associated with calibration can be reduced by the accumulation of as many counts as practical during the calibration process. The two controllable factors for achieving this are the amount of activity in the calibration source and the counting time allocated for the calibration. As a general rule, at least 10,000 counts should be accumulated during the counting of the calibration source. This may not always be practical when the activity of the calibration source must be limited for reasons listed below.

The activity of calibration sources should be limited to an amount that will not lead to significant dead-time losses and random summing in the instrument being calibrated. Unaccounted for, dead-time losses and random summing could lead to an efficiency determination that is biased and artificially low. In addition, one must be aware of the potential for detector contamination, this is particularly true for semiconductor detectors used for alpha spectrometry.

16.3 General Test Source Characteristics

The goal of test source preparation is to achieve maximum detection capability while introducing minimum bias and uncertainty into the measurement. To realize this goal, test sources must be

prepared in a consistent manner relative to the geometry, disposition of test source material, and the source container.

16.3.1 Geometrical Arrangement

The geometry of a test source must be suitable for the counting instrument and—particularly—it must be reproducible. The radioactivity associated with test sources is measured in geometries that have been standardized by measuring the instrument response to a known quantity of radioactivity in the identical geometry as the calibration source, to the extent possible. Thus, for this standardization to be accurate over time, the test source geometry must remain constant from source to source and with respect to that of the calibration source. This requirement is necessary for performing quantitative and unbiased measurements of all types of radioactivity and for all types of measurement instruments.

16.3.2 Uniformity of Test Source Material

Test source uniformity is related to the physical nature of the source material. Uniformity of test source material relative to its thickness, density (which can be influenced by water content), and homogeneity is important. Nonuniformity can result from a variation in the thickness of the test source material over its cross sectional area. If test sources are deposited in a nonuniform manner, absorption characteristics will vary from source to source and acceptable reproducibility may not be achieved.

Variation in test source thickness or density can have a particularly large effect in the measurement of alpha-particle activity and, because of their smaller mass and charge, a lesser effect in the measurement of beta-particle activity. Alpha and beta test sources, once prepared, often are stored in a desiccator to maintain a constant moisture content. Test source uniformity is relevant to gamma-ray measurements, not because of the absorption of gamma-rays, but because nonuniformity (non-homogeneity) in the distribution of activity throughout a large source changes the effective detection efficiency. For example, if the gamma-ray emitting radionuclides are concentrated in the portion of the test source container nearest the detector, the counting efficiency will be greater than if the radionuclides were uniformly distributed throughout the test source. Thus, test source uniformity can have a large influence on the counting efficiency by which the activity is detected and measured. Measurements of nonuniform sources are not reproducible; thus, radioactive sources of all types must be homogeneous.

16.3.3 Self-Absorption and Scattering

Absorption and scattering within the source material are less important when measuring gamma rays than when analyzing for charged particles. Particulate activity emitted in a source can be scattered by elastic and inelastic collisions with nuclei of the source material, degrading the energy of the particle (self-scatter) or—if sufficiently thick—the particle may be absorbed totally by the source (self-absorption). A scattering/self-absorption factor can be used, however, to correct the measured activity to that of an infinitely thin source. For beta counting, this factor is proportional to $(1 - e^{-\mu x})/\mu x$, where μ is the linear absorption coefficient for beta particles in the test source material and x is the source thickness (Friedlander and Kennedy, 1955, p. 278).

Because of the much smaller mass of beta particles, scattering is more pronounced in sources emitting beta particles than in those emitting alpha particles. Depending on counter geometry, measured beta activity can first increase as the source thickness increases, because of the scattering of electrons out of the source plane and into the detector (Friedlander and Kennedy, 1955, pp. 276-278). At greater thicknesses, self-absorption begins to predominate, and the activity eventually approaches a constant value. When this occurs, the source is said to be "infinitely thick." Counting a source at infinite thickness refers to a measurement made with a source thickness such that further increasing the amount of material added would have no effect on the count rate. The minimum source thickness required for this type of measurement clearly is not more than the maximum range R of the particle in the source material, and is often estimated to be 0.75R (Friedlander and Kennedy, 1955, p. 278).

To assure that scattering does not lead to bias in test source results, it is important that standard sources prepared for determination of counting efficiency and self-absorption corrections are prepared identically in all aspects that affect absorption to test sources whose activities are to be assayed.

Self-absorption increases with the density of the source material and with the size and charge of the emitted particle. Thus, source thickness is of greater concern for measuring alpha particles than for beta-particle emissions and has even less importance in measuring gamma rays, except for low energy x- or gamma rays. Thus, test sources prepared for alpha-particle measurements must be very thin and uniform for maximum detection capability and reproducibility.

The moisture content of the source material will affect the density of the source and the absorption characteristics of the source. A change in source moisture content will alter the density and affect the reproducibility of the measurement. Thus, the amount of moisture within

the test source should be controlled. The following procedures often are followed in order to maintain a low and constant moisture content of test sources to be counted.

- Test sources prepared by coprecipitation are dried by washing the precipitate first with ethyl alcohol and then with acetone while in the filtering apparatus. Suction to the filter apparatus is continued until the test source is dry. The filter with test source is removed from the filtering apparatus, mounted on a planchet, and stored in a desiccator prior to counting.

- Electroplated test sources are dried by heating on a hot plate, in an oven, or under a heat lamp, and then stored in a desiccator until cool and ready to count.

- Laboratory samples analyzed nondestructively are usually dried prior to measurement in order to control moisture content and help ensure that test source characteristics are reproducible. Laboratory samples, such as soil, biota, vegetation, etc., are usually dried in an oven. Test sources not counted immediately, including those for gross alpha and beta measurements, as well as for gamma-ray spectroscopy, should be desiccated to maintain a constant moisture content.

- Evaporated test sources also are stored in a desiccator, after flaming, to maintain a constant moisture content.

Another concern in measuring both alpha and beta particles from deposited test sources is back-scattering: the scattering of particles from the source-mount back through the test source material and into the sensitive part of the detector. Back-scattered beta particles have degraded energies, but can have the apparent effect of increasing the counting efficiency. This may seem to have the desired effect of improving the overall counting efficiency; however, the percent of back-scattered beta particles from the test source must remain constant and be identical to that of the standard source. The magnitude of backscatter is dependent on the beta-particle energy and the thickness, density, and atomic number of the backing material (Faires and Boswell, 1981, p. 220-222). Thus, to reduce the effect of backscatter on beta-particle measurements, the test source often is mounted on a thin, low Z (atomic number), low density material, as for example aluminum foil or thin organic films (Blanchard et al., 1960). For very precise measurements, a conducting metal film is vaporized onto the organic film so that any electrical charge build up due to the emission of charged particles can be eliminated.

As with absorption, backscatter increases with the thickness of the scattering material up to a saturation level, beyond which it remains constant. The saturation level is reached at a thickness that is about one-third the maximum range of the scattered particle (Faires and Boswell, 1981, p.

221). Therefore, due to the dependency of backscatter on atomic number and thickness, the backing used for the standard source must be identical to that used for the test source mount. For example, if the presence of HCl in the test source requires changing from an aluminum planchet to platinum, a platinum backing must also be used in counting the standard source.

16.3.4 Counting Planchets

A wide variety of planchets made of platinum, nickel, aluminum, and stainless steel can be obtained in various sizes. It is normally not of great importance which type is used as long as several factors are considered (PHS, 1967, p. 20). Some factors that should be considered in selecting a planchet are:

- *Chemical reactivity*. The metal planchet must be inert to the chemicals in the test source, as corrosion of the planchet surface radically alters test source absorption and geometry characteristics.

- *Radioactivity*. The metal comprising the planchet should contain minimal radioactivity and, although this is generally not a serious problem, the planchet background shall be measured.

- *Size*. Two-inch planchets (assuming the detector is at least that large) are often preferred for gross alpha/beta counting to expedite and simplify the evaporation of liquid samples and provide a greater surface area for solid samples, while 1-inch planchets are generally used for alpha spectrometry test samples.

- *Cost*. Platinum planchets should not be used if stainless-steel ones are adequate for the purpose.

It is usually impractical to reuse planchets, and it is generally not recommended. Except for those made of platinum, planchets are inexpensive, and it is not cost effective to clean the planchets and insure they are not contaminated from the prior test source. Platinum planchets are quite expensive and usually can be cleaned effectively in acid and recounted prior to reuse to insure that they are not contaminated.

16.4 Test Source Preparation and Calibration for Alpha Measurements

Several types of instruments are used for counting alpha particles (Chapter 15, *Nuclear Counting Instrumentation*). Each type of instrument has characteristics that affect preparation and mounting of sources. Similarly, these characteristics also affect the calibration of the instrument.

This section discusses the attributes of commonly used instruments and their effects on test source and standard source preparation.

16.4.1 Proportional Counters

Proportional counters (Section 15.2.2.1) often are used to measure alpha particles, particularly when gross analyses are desired. Proportional counters may be "internal," where the test source is placed into the detector or "windowed," where a thin window covers a part of the detector and separates the source from the detector.

16.4.1.1 Alpha Test Source Preparation

Test sources for proportional counters are usually prepared by electrodeposition, coprecipitation, or evaporation, as described below in Section 16.7.6. For internal counters, since the source is placed within the detector, care must be exercised in test source preparation to avoid the inclusion of chemicals which may react with the detector materials. Likewise, any spillage of test source material can result in contamination of the detector.

The absorption of alpha particles in the source material (self-absorption) is quite important when using proportional counters, or other ionization counters, and must be addressed when preparing a test source for counting. Self-absorption is primarily a function of source thickness (t_s) and the range (R_s) of the alpha particles in the source material. For a uniformly thick source, the fraction of alpha particles absorbed by the source increases proportionately to $t_s/2R_s$, when $t_s < R_s$ (NCRP, 1978, pp.104-105). Thus, to approach absolute counting in either 2π or 4π counting geometries, test sources should be prepared as thinly and uniformly as possible.

Another method sometimes used for alpha-emitting test sources in ionization counters is to perform the count at infinite thickness (Section 16.3.3). The count rate of a test source at infinite thickness usually is related to the count rate of a standard source prepared and measured in the exactly the same manner.

Backscatter from alpha sources increases with the atomic number of the backing or source material and with decreasing alpha energy (NAS/NRC, 1962, p. 115). Scattering of alpha particles from the source material itself is not a significant problem, and scattering from the source backing has only a small affect for very thin sources (NCRP, 1978, p. 107). When stainless-steel planchets are used, the increase in a count rate because of alpha backscatter is only about 2 percent (PHS, 1967, p. 19).

16.4.1.2 Proportional Counter Calibration — Alpha

Calibration sources prepared for calibrating counters for a specific nuclide measurement shall contain a radionuclide of similar alpha energy and be measured under identical conditions as the test sources to be measured (ASTM D3648). A variety of radionuclides have been recommended for calibrating for gross alpha analyses (Table 16.1).

TABLE 16.1— Nuclides for alpha calibration

Purpose	Nuclide	Reference
Specific Nuclide and Gross Alpha	^{239}Pu, ^{241}Am, ^{210}Po, ^{228}Th, ^{226}Ra, ^{233}U, ^{235}U, and U_{nat}	ASTM D3648
Gross Alpha	^{241}Am	EPA ,1980
Gross Alpha	^{241}Am, ^{237}Np, and U_{nat}	ASTM D1943
Gross Alpha	^{241}Am, ^{239}Pu, ^{230}Th, and U_{nat}	APHA (1995), Method 7110

To the extent possible, standard sources should be prepared in a manner identical to the method used for test source mounting. The counting efficiency (ε) is then determined by counting the standard source for a sufficient time to accumulate approximately 10,000 counts and dividing the derived counts per second (cps) by the α emission rate of source in disintegrations per second (dps).

$$\varepsilon = \frac{cps}{dps}$$

In cases where finite test source thicknesses are unavoidable, alpha-source counts can be adjusted to account for self-absorption (PHS, 1967, p. 19). This requires that a self-absorption curve be prepared in order to determine the change in counting efficiency as a function of source thickness or mass. Standard sources containing a known amount of the radionuclide of interest are prepared in varying thicknesses (mass) and counted. Absorption curves for gross alpha-particle measurements most often are constructed using reference material containing one of the nuclides listed above. The absorption curve is constructed by counting planchets containing varying mass of material but with constant added radioactivity. A curve is generated by plotting the efficiency at a given source thickness divided by the efficiency at "zero" thickness versus source mass (mg) or density thickness in $\mu g/cm^2$ or mg/cm^2 (NCRP, 1978, p. 105). Thus, the efficiency relative to the "zero thickness" efficiency can be read directly from this curve for any measured test source thickness. Test sources prepared for gross measurement are counted in the exact geometry as those used to prepare the absorption curve. The material forming the matrix for the self-absorption standard source should, when possible, be identical to that expected in the test sources to be analyzed. Based on the test source mass or density thickness in units of $\mu g/cm^2$ or mg/cm^2,

the correction factor determined from the absorption curve is applied to the test source count, yielding the count rate equivalent to an infinitely thin source.

Most modern proportional counters are capable of simultaneous alpha and beta counting. This is accomplished by identifying the two types of particles based on their pulse height. Those pulses whose heights exceed an experimentally established discriminator level are registered as alpha counts and those falling below this level are recorded as beta counts. Some fraction (usually less than 10 percent for a weightless source) of the alpha particles is recorded as betas, even for nearly weightless test sources. This fraction increases as the thickness (mass) of the source increases. A much smaller (often insignificant) fraction of the beta interactions are registered as alphas. This misclassification of alpha and beta counts is referred to as "crosstalk."

For simultaneous alpha and beta counting, corrections must be made to the beta count rate to remove the portion contributed by alpha particles. Since the fraction of alpha counts occurring in the beta channel is a function of the source mass, a crosstalk curve relating the fraction of alpha particles counted as beta to source mass must be developed. This can be accomplished concurrently with the self-absorption calibration if the radionuclide selected is an alpha emitter only—no beta particles. This is done by recording the beta counts from the alpha self-absorption determination at all source weights and plotting the fraction (beta counts/alpha + beta counts) as a function of source mass (Section 17.4.1). Beta count rates then can be corrected for the influence of the alpha particles at all source thicknesses.

16.4.2 ZnS(Ag) Scintillation Counter

This type of counter is discussed in Section 15.2.2.3. Because the alpha particle must be emitted from the source and interact with the screen, as it does with the ionization chamber of an internal proportional counter, the previous description concerning self-absorption and scatter of alpha particles during analysis in an internal proportional counter may be applied to counting alpha particles with a ZnS(Ag) scintillation counter. Additional advantages of this counting arrangement are the very low backgrounds that are achievable and the small potential for permanently contaminating the counter, because the zinc sulfide screens can be replaced.

A source mount shaped like a washer, with one side enclosed with a transparent ZnS(Ag) screen, is an arrangement often used. The test source to be counted is placed in the hole of the "washer," in contact with the ZnS(Ag) screen. The other side of the test source mount is sealed, generally with wide transparent tape, securing the test source within the source mount. The test source is then placed on an appropriately sized photomultiplier tube and counted. Because of the

availability of large photomultiplier tubes, sources up to 5 inches in diameter can be prepared for measurement (PHS, 1967, p. 26).

The considerations related to alpha calibrations, discussed above under proportional counters, apply equally to scintillation counter calibration.

16.4.3 Alpha Spectrometry With Semiconductor Detectors

Semiconductor detectors for alpha particle counting are discussed in Section 15.2.2.5. Alpha-energy spectra of very high resolution are attainable with semiconductor detectors if the prepared test source is essentially weightless, ≤ 1 µg/mm^2 (Herpers, 1986, pp. 143-145). As the thickness of the test source increases, the spectral energy is degraded due to self-absorption, which broadens the peak and forms a "tail" on the lower-energy side (Chapter 17). The alpha-energy spectral degradation will increase, as the source thickness increases, raising the possibility of overlapping peaks with a loss of spectrum integrity. Thus, it is of utmost importance to prepare very thin and uniform alpha test sources for spectrometry. This may be accomplished by electrodeposition or coprecipitation (ASTM, D3084), if reagents are controlled so that only small (milligram) quantities of precipitate are recovered (Sections 16.6.1 and 16.7.2). For example, in the coprecipitation of actinide test sources for spectral analysis, source thicknesses of 0.4 to 1 µg/mm^2 (0.04-0.1 mg/cm^2) are routinely achieved, which is quite adequate for producing well-defined alpha spectral peaks (EPA, 1984a).

Semiconductor detectors used for alpha spectrometry require both efficiency and energy calibrations. Calibration sources, traceable to NIST, often are prepared with multiple radionuclides so they may be used for both types of calibration (ASTM D3084). Sources containing ^{234}U, ^{238}U, ^{239}Pu, and ^{241}Am have been used for this purpose. When mixed-nuclide calibration sources are used, the average counting efficiency is often calculated using the efficiencies of the individual radionuclides. Some alpha spectrometry analysis programs calculate an average efficiency where the individual radionuclide efficiency is weighted by the uncertainty in its determination. Other radionuclide combinations may be used, but in addition to the requirement for traceability for the disintegration value, the energies of the radionuclides must be known with a high degree of certainty.

Calibration sources may be prepared by either electrodeposition or coprecipitation. Due to their durability and stability, electrodeposited calibration sources are often chosen. It is important that the area of deposition be consistent with that of test sources to be counted and that there are no significant impurities present (ASTM D3084). See the additional discussion on alpha spectrometer calibration in Section 17.3.2.

16.4.4 Liquid-Scintillation Spectrometer

With proper scintillators, liquid scintillation can be used to measure alpha-particle emitters (Passo and Cook, 1994) (Section 15.2.2.4). Although the relatively high background of liquid scintillation counting restricts the sensitivity relative to other counting techniques, e.g., internal proportional counting or the use of ZnS(Ag) screens, the ease of source preparation and the nearly 100 percent counting efficiency are advantages often exploited (Hemingway, 1975, p. 146). The separation of alpha- and beta-particle counts attained in the spectrometer can be enhanced by proper scintillator choice. Ultima Gold AB™ was designed specifically to maximize alpha/beta separation in aqueous solutions and, in other studies, poor alpha/beta separation has been overcome by making the standard cocktail 20 percent in naphthalene (Passo and Cook, 1994, pp. 3-11 to 3-12). It is believed that naphthalene improves the alpha/beta separation by acting as an intermediate in the energy transfer process between the solvent and the fluor (McDowell, 1986).

EPA's (1978) recommended procedure for measuring ^{222}Rn in water uses liquid scintillation counting. The protocol is based on the solubility of radon in a number of scintillators. To measure radon in air, the radon is first adsorbed onto activated charcoal and then mixed with an appropriate scintillator and counted (EPA, 1987; Passo and Cook, 1994, pp. 8-5 to 8-10). Utilizing the high solubility of ^{222}Rn in organic solvents, concentrations of ^{222}Rn in air have been determined by bubbling air through the scintillator in a scintillation vial (Amano et al., 1985). Concentration of ^{222}Rn, determined by liquid scintillation, also can be used in the measurement of its parent, ^{226}Ra.

Some actinides (U and Th) and transuranics (Np, Pu, Am, and Cm) have been measured by a procedure that involves "Extraction Scintillation Techniques" (Passo and Cook, 1994, pp. 6-1 to 6-2 and 13-1 to 13-6). An extraction agent, e.g., bis(2-ethylhexyl) phosphoric acid (HDEHP), is mixed either with a toluene or a di-isopropylnaphthalene (DIN) based cocktail. The alpha emitter, in the aqueous laboratory sample, is extracted into the scintillation mixture and counted by liquid scintillation. The discussion in Section 16.5.2.1 can be applied to both alpha and beta particles.

16.5 Characteristics of Sources for Beta Measurements

16.5.1 Proportional Counters

Beta decay generally is accompanied by gamma-ray emission; the latter normally is much easier to identify and quantify. Beta-particle counting typically is more difficult, due to the additional

source preparation and associated complications resulting from the effects of backscatter, scattering, and absorption in the source material (NAS/NRC, 1962, p. 118-119). Beta particles are not emitted monoenergetically and may result in additional difficulty in quantitative measurements.

Beta counting in ionization-type counters often is used after chemical separations are performed to isolate the beta-emitting radionuclide of interest from other radionuclides. Beta measurements are performed on chemically isolated pure beta emitters (beta decay not accompanied by a gamma-ray) and also in cases when increased sensitivities are required to meet detection limits, such as, ^{89}Sr, ^{90}Sr, ^{99}Tc, ^{131}I, ^{134}Cs, and ^{137}Cs (EPA, 1980). The proportional counter often is used for measuring these beta-particle emitters. Test sources measured in a proportional counter are usually prepared by electrodeposition, coprecipitation, or evaporation, as described below in Section 16.7 (Blanchard et al., 1960). The comments on chemical reactivity of source contained materials and contamination given in Section 16.3.1, apply here.

16.5.1.1 Beta Test Source Preparation

Although it remains a consideration, self-absorption of beta particles is not as pronounced as with alpha particles, because the charge and mass of beta particles are significantly smaller. Scattering, and particularly backscatter from the source mount, is much more pronounced for beta counting than for alpha counting (Blanchard et al., 1957). To reduce scatter, plastic mountings are often used to mount sources for beta counting (EPA, 1980). The effects resulting from self-absorption and scattering can be minimized by preparing test sources in a standardized constant thickness, or using a correction factor based on an empirical calibration curve for different thicknesses (Friedlander and Kennedy, 1955, pp. 276-277; Tsoulfanidis, 1983, pp.133-134). (Section 16.3.3.)

For sufficiently thick sources, the beta particles emitted from the source reach a limit, and the count rate becomes independent of the source thickness.

16.5.1.2 Proportional Counter Calibration — Beta

As in other calibrations, proportional counters used for beta-particle analysis shall be calibrated with NIST traceable standards in a manner that is totally consistent with the counting of test sources. When possible, the radionuclide to be quantified should be used as the calibration source. For gross beta analysis, the radionuclides presented in Table 16.2 have been recommended for calibration sources.

TABLE 16.2 — Nuclides for beta calibration

Purpose	Nuclide	Reference
Gross Beta	^{137}Cs	ASTM D3648
Gross Beta	^{137}Cs	EPA, 1980
Gross Beta	^{137}Cs	ASTM D1890
Gross Beta	^{137}Cs and ^{90}Sr-^{90}Y	APHA (1995), Method 7110

If test sources of varying mass are to be counted for beta activity determination, a self-absorption curve must be prepared. The method used is identical to that described under alpha calibration for proportional counters, except that a beta-emitting reference material is used instead of alpha.

16.5.2 Liquid-Scintillation Spectrometers

When beta measurements are required, especially those involving pure beta emitters of low energy, they are often performed in a liquid scintillation spectrometer, because self-absorption and backscatter are eliminated and counting efficiencies are relatively high (Herpers, 1986, pp. 133-135). Although it is the preferred instrument to measure low-energy, pure beta-emitting radionuclides, e.g., ^{3}H, ^{14}C, and ^{35}S, it is a well-established procedure for measuring numerous other beta-emitting radionuclides, including ^{45}Ca, ^{65}Zn, ^{141}Ce, ^{60}Co, ^{84}Sr, ^{55}Fe, ^{87}Rb, ^{147}Pm, and ^{36}Cl (Hemingway, 1975, pp. 145-146). The liquid scintillation spectrometer, applied to beta-particle measurements, is described in detail in Section 15.3.3.

Tritium is the radionuclide most often measured by liquid scintillation counting (DOE, 1997; EPA 1979; Lieberman and Moghissi, 1970, p. 319). The primary step in preparing water samples for counting is distillation in the presence of an oxidizing agent, such as $KMnO_4$, to separate the tritium labeled water from dissolved solids, including interfering radionuclides, and any organic material that may be present. An aliquant of the distillate is then mixed with a liquid scintillator and counted in a liquid scintillation spectrometer. To measure tritium in samples of other matrices, the water in the sample can be removed and collected by distillation as an azeotrope with, for example, *n*-hexane or cyclohexane (Moghissi, 1981; EPA, 1979). An aliquant of the water collected is then mixed with a liquid scintillator and counted, as described above for water samples.

Tritium can be concentrated in a sample of water if lower detection limits are required. The concentration process, electrolysis, uses the isotopic effect caused by the large mass difference (three times) between ^{1}H and ^{3}H (DOE, 1997; EPA, 1984a). Tritium becomes enriched as electrolysis continues. Generally, 50 mL of the laboratory sample is placed in an electrolysis cell and a current of about three amps applied. Electrolysis is continued until the volume reaches

about 5 mL. More sample can be added to the cell during the electrolysis, if greater sensitivity is necessary for the measurement. The concentrated laboratory sample is then distilled in the presence of an oxidizing agent, such as $KMnO_4$, and treated like a water sample (see above).

16.5.2.1 Liquid Scintillation Test Source Preparation

The preparation of a laboratory sample for a liquid-scintillation spectrometer usually is relatively simple and fast. The radionuclide to be measured is isolated in a solution, which is then introduced into and thoroughly mixed with one of a variety of ready-to-use commercially available liquid scintillators. This mixture is often referred to as a scintillation "cocktail." The liquid scintillator is an emulsion system, usually consisting of an aromatic solvent containing the appropriate scintillator mixed with a detergent (NCRP, 1978, pp.168-169). If a sample is insoluble in the scintillator, it can be ground to a fine powder, stirred into the scintillator until a homogeneous mixture is formed, and solidified with a gelling agent (Friedlander et al., 1981, p. 303).

Because much or our ecosystem consists of materials composed of carbon and hydrogen, the measurement of ^{3}H and ^{14}C levels in biological materials is important. Water, for ^{3}H analysis, can be recovered efficiently from all types of environmental and biological samples by azeotropic distillation. The laboratory sample is distilled with a hydrocarbon, such as benzene or cyclohexane, which is compatible with the liquid scintillation process (Moghissi et al., 1973; Moghissi, 1981). The distillate is mixed with the proper scintillator and counted in a liquid scintillation counter. Tritium has been successfully measured by this technique in such samples as animal and human tissues, soil, hay, grass, urine, and milk.

Environmental and biological samples also can be analyzed for total ^{3}H (that contained in both the water and fibrous fractions) by quantitatively combusting the laboratory sample, collecting the water formed, and analyzing it by liquid scintillation spectrometry (DOE, 1997). In another case, both ^{3}H and ^{14}C can be measured simultaneously (EPA, 1984b). The laboratory sample first is freeze-dried to remove and collect the water fraction. The tritium in the water is measured directly by liquid scintillation spectrometry. The fibrous (freeze-dried) material is combusted and the H_2O and CO_2 are collected. As before, the ^{3}H in the water is measured directly by liquid scintillation spectrometry, while the ^{14}C is first converted to benzene or captured as CO_2 and then counted by liquid scintillation spectrometry.

A primary problem with measurements using a liquid-scintillation spectrometer is "quenching." Quenching occurs when the production of light is inhibited or the light signal is partially absorbed during the light transfer process by a substance in the liquid. The two basic types are

chemical and color quenching. Some of the stronger chemical quenchers are alkyl bromides, iodides, nitrates, mercaptains, and ketones (NCRP, 1978, p. 46). Color quenching involves the reduction of light transmission through the solution to the cathode of the phototube by the absorption of the light photons. The two techniques most often used to correct for quenching involve the use of internal or external standards.

Chemiluminescence, the production of light by a chemical reaction, can be troublesome in liquid-scintillation counting. However, the duration of chemiluminescence is generally short, and a wait of a few minutes after mixing the reagents will allow the effect to dissipate before counting starts. Similarly, phosphorescence, the emission of light from certain chemicals caused by exposure to light, will cease a short time after being placed in the dark. This is referred to as "dark adapted" (Faires and Boswell, 1981, p. 182).

16.5.2.1 Liquid-Scintillation Spectrometer Calibration

When the quenching of a group of test sources is predictable, e.g., distilled drinking water (EPA, 1980; ASTM D4107), a counting efficiency is determined for the group by placing a known quantity of reference material in the source medium and scintillation solution under identical conditions (vials and volumes) as the sample medium.

Except for test sources with very predictable amounts of quenching, it may be necessary to determine a counting efficiency for each laboratory sample. Two methods of determining counting efficiency are available: internal standardization and external standardization (NCRP, 1978).

Internal standardization for quench correction is by the method of standard additions. This involves the counting of two aliquants of a sample, one being the sample and the other is an identical aliquant that has been spiked with a known amount of the radionuclide being determined. The degree of quench can then be determined from the spiked aliquant and applied to the unspiked sample (DOE, 1995). This method does not require a curve for correction but decreases throughput because two test source counts are required. For these reasons, the use of an external standard is the more widely used technique to correct for quenching (Horrocks, 1973).

One external standard method is also called the "external-standard channels-ratio" (Baillie, 1960; Higashimura et al., 1962). In this method, a series of vials is prepared containing a known amount of reference material and varying amounts of the medium being evaluated. Windows in the energy spectrum are set for a high- and low-energy region. The vials are counted and the ratios of low-to-high count rates are recorded for each quenched source. A quench curve is then

prepared by plotting the ratios of low-to-high energies as a function of counting efficiency. The efficiency of an unknown test source can then be determined from its low-to-high energy ratio during counting.

The second external-standard method employs an external gamma-ray source that generates Compton electrons in the scintillation solution. Count rates from the external source are determined for a set of sources whose efficiency is known from the internal-standard method. A quench curve is then prepared by plotting the external count rate vs. counting efficiency.

The external-standard methods should not be generalized beyond use for the media conditions under which they were prepared.

16.6 Characteristics of Sources for Gamma-Ray Measurements

Backscatter and self-absorption, which must be addressed when measuring alpha and beta emissions, cause less uncertainty in the measurement of most gamma-ray emitters. This is because the penetrating nature of gamma rays is totally different from that of particles. For thick samples or high-Z matrices, a detection-efficiency correction is necessary for low-energy photons (especially below 200 keV) due to the self-absorption of photons in the sample. There is, however, some backscatter of gamma-rays from the shield surrounding the detector, which produces a small peak at about 200 keV (NAS/NRC, 1962, p. 32).

16.6.1 Gamma Test Source Preparation

No significant precautions usually are required in preparing test sources for gamma-ray spectrometry, as long as the test source is homogenous and positioned reproducibly relative to the detector. Although source properties (e.g., density and moisture content) are not as important in gamma-ray spectrometry as in alpha or beta measurements, test source preparation for gamma measurements may still include drying and ashing to control moisture content and to reduce the test source size. Homogeneity of the test source can be attained by thoroughly mixing laboratory samples that have been ashed (many combustible matrices not containing volatile radionuclides are ashed), by grinding and mixing solids (e.g., soils and sediments), or by finely chopping and mixing fresh vegetation. Also, calibrations are generally conducted using standard sources with identical counting geometries and the same or similar matrices as the test source for analysis.

Important considerations in preparing test sources for gamma-ray spectrometry are geometry (shape), size, and homogeneity (uniformity) of the source. Test sources can be in any reproducible shape or size, but the radionuclides must be uniformly distributed throughout. A

counting container that allows the source to surround the detector, thus maximizing the geometrical efficiency, is referred to as the "Marinelli" or "reentrant" beaker (Hill et al., 1950). It consists of a cylindrical sample container with an inverted well in the bottom of the beaker that fits over the detector.

Counting efficiencies are determined by measuring a known quantity of the radionuclide(s) of interest in the same matrix and source-detector configuration as the sources requiring analysis (NCRP, 1978, pp. 243-244; ASTM, D3649). This eliminates any effect that might be caused by differences in test and calibration source characteristics, e.g., density, moisture content, shape, and size. Efficiency curves may be prepared for a detector by measuring a variety of standardized sources having different photopeak energies under identical conditions as the unknown (Coomber, 1975, p. 18; ANSI, 1991).

Two important advantages of gamma-ray spectrometry are the ability to measure more than one radionuclide simultaneously and the elimination or reduction of the necessity for chemical dissolution and radionuclide separations (nondestructive analysis). Source configurations for nondestructive analyses generally are selected to optimize counting efficiency. Examples are (PHS, 1967, p. 78):

- Marinelli beakers of various volumes to measure liquid sources, as water, milk, and food samples blended to a slurry;
- Cylindrical plastic containers of various volumes, such as the 400 mL "cottage-cheese container" frequently used for containing solid sources;
- Planchets of various diameters to measure precipitates, air filters, etc.; and
- Aluminum cans of a standardized volume into which solid sources can be compressed, and sealed, if desired, to retain radon.

If greater counting efficiency is required, the test source size can be reduced, allowing a greater amount of the laboratory sample to be counted and in a more favorable geometry. Examples of such processes are:

- Reducing the volume of water samples by evaporation;
- Reducing the volume of water samples by co-precipitating the desired radionuclides;

- Reducing the size of vegetation samples by compression into a large pellet or by ashing, if volatile radionuclides are not of interest; and

- Reducing the size of filter samples by compressing the sample into a reduced standard volume or by ashing, if volatile radionuclides are not of interest.

16.6.2 Gamma Spectrometer Calibration

Most gamma-ray spectrometry systems are calibrated with either single or mixed standards in an exact matrix and geometric form as the samples to be analyzed. However, there are computer codes that can calculate detector efficiency from the physical dimensions of the detector and sample counting geometry (Mitchell, 1986; Hensley et al., 1997).

Commercial standards of single or mixed gamma-ray emitters in a matrix of known chemical composition and density can be prepared in user-supplied containers. Calibrations based upon these standards can then be adjusted to correct for any differences in composition and density between the calibration source and the test source (Modupe et al., 1993).

MARLAP recommends that calibration data for gamma spectroscopy calibration be obtained from the National Nuclear Data Center at Brookhaven National Laboratory (http://www.nndc.bnl.gov/nndc/nudat/). Calibration data are readily available for common radionuclides, including ^{210}Pb, ^{241}Am, ^{109}Cd, ^{57}Co, ^{141}Ce, ^{139}Ce, ^{203}Hg, ^{51}Cr, ^{113}Sn, ^{85}Sr, ^{137}Cs, ^{54}Mn, ^{88}Y, ^{65}Zn, ^{60}Co, and ^{40}K. For more information on gamma spectrometry calibration see ANSI 42.14. (Also see Section 17.3.1.6 on gamma calibration.)

16.7 Methods of Test Source Preparation

16.7.1 Electrodeposition

High-resolution spectroscopy requires a very thin, uniform, flat, and nearly weightless source mount. Ideally, the source plate to determine alpha activity by a spectrometer would be a flat plate coated with a monolayer of radioactive atoms and with no foreign material above the layer to attenuate the alpha radiation (Kressin, 1977). The electrodeposition of radionuclides on a suitable metallic surface from an aqueous solution often can produce thin and uniform test sources that approach these ideal conditions. Thus, this technique is very appropriate for preparing sources of alpha emitters, especially the actinides, which include uranium, plutonium, thorium, americium, and neptunium (ASTM, D3865; DOE, 1997; EPA, 1979).

There are a number of electrolytic cell designs used to electrodeposit radionuclides. The cathode, on which the radionuclide deposits is often a thin metal foil or disc, such as platinum or stainless steel, or a metal-coated plastic film (Blanchard et al., 1960). The stirring rod, often made of platinum, can also serve as the anode of the cell. Deposition of actinides for alpha spectrometry also has been performed on disposable cells constructed form 20 mL polyethylene scintillation vials and highly polished stainless steel planchets (Talvite, 1972). Disposal prevents cross contamination. The composition of the electrolyte and the parameters applied in the electro-deposition process, such as applied voltage, amperage, current density, and deposition time, are dependent upon the chemical properties of the element, especially its reduction potential, and foreign material that might be present. Thus, "Each element requires optimization of its own procedure" (Adloff and Guillaumont, 1993, p. 158). Deposition time varies from 10 minutes to two hours.

Actinides and similar elements are extremely hydrolytic and can deposit on the glass cell wall or anode or precipitate during deposition (Puphal et al., 1983). Electrodeposition typically is performed, therefore, in electrolytic solutions at low pH (≈2) to prevent hydrolysis or precipitation. The solution may contain complexing agents (such as fluoride) and chelates (such as EDTA) to minimize the effect of interfering ions, commonly encountered in biological and environmental samples (Puphal and Olsen, 1972). The procedure of Kressin (1972), however, illustrates the admonition of Adloff and Guillaumont cited above: citrate and fluoride, a chelate and complexing agent, respectively, each interferes with the electrodeposition of plutonium and americium in his process.

Electrodeposition is applicable to more than 30 radionuclides. The main advantage of electrodeposited sources over those from other methods of preparation is their extremely thin, uniform deposit of a radionuclide on a plate, which permits high resolution spectroscopy; however, the yield is often not quantitative (Adloff and Guillaumont, 1993, p. 158). Thus, the yield must be monitored with the inclusion of a known quantity of an isotope, which is deposited simultaneously with the analyte. Radioactive sources of the following elements have been prepared successfully by electrodeposition (DOE, 1997; Blanchard et al., 1960; Johnston et al., 1991.)

Actinium	Gold	Polonium	Strontium
Americium	Hafnium	Promethium	Tellurium
Antimony	Indium	Protactinium	Thallium
Bismuth	Iron	Radium	Thorium
Cadmium	Lead	Rhenium	Tin
Cobalt	Neptunium	Ruthenium	Uranium
Copper	Nickel	Selenium	Yttrium
Curium	Plutonium	Silver	Zinc

Particularly important to environmental analysis is a procedure by which virtually all alpha-emitting nuclides—radium through californium—can be determined in soil in any combination on a single sample with few interferences using electrodeposition to prepare the source (Sill et al., 1974).

Although sources of radioactive isotopes of these elements have been prepared by electro-deposition, it might not be the preferred technique in some of the examples cited. For various reasons, other methods of test source preparation may be superior: yields can be low, the presence of other metals sometime interferes, the quality of deposition might be poor (flaking), the recovery can be low, the spectral resolution might be poor, and some procedures require rather elaborate equipment, are expensive, and are time consuming, thus labor intensive (Sill and Williams, 1981; Hindman, 1986). Interference will be caused by several factors: (1) "Any element present in the separated fraction that is able to be electrodeposited will be present on the metal disc;" (2) "Incomplete separation of rare earth elements or incomplete wet ashing for the removal of organic material will decrease the efficiency of the electrodeposition and may result in a thick deposit unsuitable for α-spectrometry measurement;" and (3) "Samples containing more than 20 µg of U are unsuitable for measurement by α spectrometry due to the thickness of the deposit" (DOE, 1997, p. 4.5-270). When stainless-steel planchets cannot be used, because of the corrosive nature of the electrolyte, and platinum is required, the method can be quite expensive and time consuming, since recycling of the expensive electrode material requires thorough cleaning to prevent cross contamination.

Test sources of actinides are often prepared by electrodeposition with yields of 90 percent and higher (DOE, 1997; EPA, 1979; Sill et al., 1974; Puphal and Olsen, 1972; Kressin, 1977; Talvite, 1972; Mitchell, 1960; Shinohara and Kohno, 1989, pp. 41-45). In addition, ^{54}Mn sources have been successfully prepared by the electrodeposition from mixed-solvent electrolytes onto stainless steel planchets (Sahoo and Kannan, 1997, pp. 185-190).

If the redox couple between the metal cathode and the radionuclide to be deposited is positive, the radionuclide will deposit spontaneously. That is, it will deposit quantitatively without using any applied potential. Generally, a metal planchet is simply suspended in the solution that is stirred with a glass stirring rod for a few hours (Blanchard, 1966; DOE, 1997). An example of such a spontaneous reaction between polonium and nickel is given below.

$$Po^{+4} + 2\ Ni \rightleftharpoons Po + 2\ Ni^{+2} \qquad E^{o} = 0.98\ \text{Volt}$$

Polonium also will deposit quantitatively on silver planchets. ^{210}Po is an important naturally occurring radionuclide that is often included in environmental studies. Spontaneous deposition

onto either nickel or silver is the preferred technique for preparing ^{210}Po sources for measurement.

A similar technique, called internal electrolysis, is preformed by selecting electrodes that have a large difference in potential. A conventional electrolytic cell containing an acid solution of the radionuclide to be deposited may be used. A magnesium (E^o = +2.37 volts) strip, for example, is inserted into the electrolyte and connected by an external circuit to the inert metal cathode (planchet), usually platinum. A spontaneous current flows and deposition on the cathode will occur. The conditions at the inert cathode are exactly the same as if an external voltage were applied; however, longer electrolysis times are necessary to achieve quantitative recoveries. Very thin and uniform sources of ^{106}Ru, ^{110}Ag, ^{203}Hg, ^{60}Co, ^{114}In, ^{51}Cr, ^{198}Au, and ^{59}Fe were prepared by this technique, with greater than 96 percent recovery in all cases (Blanchard et al., 1957, pp. 46-54; Van der Eijk et al., 1973).

16.7.2 Coprecipitation

Coprecipitation (Section 13.8) has been employed to mount sources for alpha spectrometry. Some radiochemists prefer the method to electrodeposition, maintaining that, "The procedure is faster and more reliable than those involving electrodeposition and gives consistently higher yields" (Sill and Williams, 1981). Hindman (1986) asserts that the method is "more rapid, more economical, and more efficient" ... "and yields good decontamination factors, high recoveries, and excellent resolution of the α spectra for uranium, plutonium, americium, and thorium."

Although sources prepared by coprecipitation are thicker than those prepared by electrodeposition, sufficiently thin sources, even for alpha spectrometry, can be prepared by controlling the amount of precipitate formed. Sources thinner than 0.5 $\mu g/mm^2$ can be prepared of the actinides by coprecipitation (EPA, 1984a). Thicker sources lead to poor resolution of the spectra (Hindman, 1983) and sources produced by any technique that are greater than 10 $\mu g/mm^2$ lead to attenuation of alpha particles (Adolff and Guiallaumont, 1993, p. 161).

After separations are completed, a slurried precipitate is poured quantitatively through a filtering apparatus collecting the precipitate on a small (e.g., 25 mm dia.) filter. Vacuum filtration often is used to speed the operation. With suction applied, the precipitate typically is washed with water, then ethyl alcohol, and finally with acetone to dry the precipitate. The filter is removed from the filtering apparatus and mounted on a metal planchet, commonly with double-stick tape, and stored in a desiccator to await counting. Any ^{222}Rn progeny that collects on the filter during the filtration process will decay in a short period of time and not affect the measurement. Samples of the following radionuclides have been prepared for quantitative analysis by coprecipitation:

Radionuclide	Carrier	References
^{32}P	$MgNH_4PO_4$	a
^{51}Cr	$BaCrO_4$	a
$^{89/90}Sr$	$SrC0_3$	a,b,c
^{90}Y	$Y_2(C_2O_4)_3$	a,b,c
^{131}I	PdI_2	a,b,c
^{137}Cs	Cs_2PtCl_6	b
^{147}Pm	$Nd_2(C_2O_4)_3$	a
^{210}Bi	BiOCl	a
^{226}Ra	$BaSO_4$	b
Th	$Ce(IO_4)_4$	d
Th	LaF_3	a,b
U	LaF_3 (NdF_3)	a,b,(f)
Np	LaF_3	b
Pu	$LaF_3(NdF_3)$	a,b,d,(f)
Am	$LaF_3(NdF_3)$	a,b,d,(f)
Cm	LaF_3	b
Th	$Ce(OH)_2$	e
Np	$Ce(OH)_2$	e
Pu	$Ce(OH)_2$	e
Am	$Ce(OH)_2$	e
Cm	$Ce(OH)_2$	e
U	UF_3	e

a	EPA (1984)	c	DOE (1997)	e	Sill (1981)
b	EPA (1980)	d	Hindman (1983)	f	Hindman (1986)

It should be emphasized that precipitated sources must be thoroughly dry before measurement, otherwise, self-absorption and scattering will change with time as water evaporates. Also, sources are often covered with a thin film, such as Mylar™ or Formvar™, to avoid sample loss and contamination of counting equipment. Care must be taken to avoid excessive handling of the source that can change the physical nature of the co-precipitate, producing an uneven thickness.

Another precipitation technique has been applied to preparing radioactive sources. Source preparation by precipitation can be conducted in a desiccator fitted with a valve to allow first the evacuation of the desiccator and then the admission of a precipitating gas, such as ammonia (NH_3) or hydrogen sulfide (H_2S) (Blanchard et al., 1957, pp. 26-31; Van der Eijk et al., 1973). A carrier is added to the sample and a know quantity is pipetted onto a planchet. The planchet containing the test source solution is placed in the desiccator and exposed to a precipitating gas for one to two hours. This period of time allows settling to occur. The test source is removed from the desiccator and evaporated beneath a heat lamp. Using an $AlCl_3$ carrier in an ammonia atmosphere, Yoshida et al. (1977) prepared uniformly deposited radioactive sources of ^{59}Fe, ^{60}Co, ^{95}Nb, ^{103}Ru, and ^{198}Au by this technique.

16.7.3 Evaporation

When a high degree uniformity of the deposit is not a requirement for the measurement, sources can be prepared by simple evaporation under a heat lamp (Bleuler and Goldsmith, 1952). This procedure is easy, fast, and adequate for many type measurements. Water samples for gross alpha and beta screening measurements are often prepared by this method (EPA, 1984a; EPA, 1980). An aliquant of the water laboratory sample is evaporated on a hot plate until only a few milliliters remain. The concentrated solution that remains is then transferred quantitatively with a pipette to a tared stainless-steel planchet, usually 2-inch diameter, and evaporated to dryness under a heat lamp. The planchet, with the evaporated test source, is then flamed over a burner until dull red to reduce the amount of solids present and to convert the matrix to an oxide. (Insoluble hydroxides, which are often bulky and gelatinous, are prime candidates for ashing, as the oxide formed is much firmer, more uniform, and better defined.) The test source is cooled, weighed, and counted for alpha and beta particles in a proportional counter. Planchets containing evaporated solids cannot be flamed if volatile radionuclides are to be measured.

Most of the solids in an evaporated source deposit in a ring around the edge. Techniques to improve uniformity include the addition of a wetting agent, such as tetraethylene glycol or a 5 percent insulin solution (Shinohara and Kohno, 1989), freeze drying the sample, or precipitation and settling of the active material prior to evaporation (Friedlander et al., 1981, p. 305; Van der Eijk and Zehner, 1977). The wetting agent is pipetted onto the spot to be covered by the test source, then removed with the pipette. That remaining can be dried under a heat lamp. A known quantity of the laboratory sample is then pipetted onto the spot and dried under a heat lamp. Additional portions of the sample may be added and evaporated.

Sample spreading on the planchet, as it is heated, can result in depositing test source material on the planchet walls or in the flow of the liquid over the edge of a flat, lipless planchet. Such spreading can be controlled or restricted by outlining the desired source area with a wax pencil. Metal planchets often are constructed with a small lip around their circumference that retains the test source on the planchet. All sources prepared by evaporation should be flamed to a dull-red color, cooled, and stored in a desiccator until counted, unless they contain volatile radionuclides, in which case simply store the evaporated test source in a desiccator.

Source spreading during evaporation has been restricted by electrospraying a silica gel suspension onto a thin film to produce a circular pad. The radioactive source solution is dropped onto the circle and evaporated to dryness (Chen et al., 1989).

EPA's (1980) prescribed Method 900.0 for measuring gross alpha and beta radioactivity in drinking water suggests that the sample aliquant be limited to what will produce 5 mg/cm^2 of solids on the planchet. Thus, for a 2-inch planchet (20 cm^2), an aliquant containing 100 mg of non-volatile dissolved solids is the recommended maximum test source mass.

After a radionuclide in solution has been purified by chemical techniques, i.e., impurities removed, the solution can be transferred to a planchet and evaporated to dryness, as described above. Evaporation of a laboratory sample after purification is used by the EPA to measure ^{228}Ac in the analysis for ^{228}Ra (EPA, 1984a), and sources of thorium, isolated from marine carbonates, have been prepared by evaporation for measurement by alpha spectrometry (Blanchard et al., 1967). Measured count rates of identified radionuclides, for which absorption curves have been prepared, can be adjusted for self absorption in evaporated test sources.

In the case of all dry sources, steps should be taken to prevent solids from exiting the planchet, which will affect the measurement and, in time, contaminate the detector. Sources consisting of loose, dry material, or with a tendency to flake, should be covered with thin plastic or immobilized by evaporating a few drops of a lucite-acetone solution on the solid deposit (PHS, 1967, p. 21).

16.7.4 Thermal Volatilization/Sublimation

Vacuum thermal volatilization or sublimation are often used when very thin and uniform sources are required (Blanchard et al., 1957, p. 7-9 and Friedlander and Kennedy, 1955, p. 122). The disadvantages of this technique are that it is time consuming and the recoveries are often less than 50 percent (NAS/NRC 1962, pp. 126-127).

The apparatus used to perform this procedure consists of a demountable vacuum chamber that contains either a ribbon filament, often with a shallow trough, or a crucible. The collector plate is usually mounted less than an inch away. The source solution is first evaporated onto the filament. As the required temperature of the filament is reached, the trough in the filament tends to collimate the sublimed material onto the collecting plate, increasing the recovery of the sample.

Pate and Yaffe (1956) designed a system for volatilizing radionuclides from a crucible heated with electrical resistance wire. Their design resulted in nearly 100 percent yields on thin collecting films, and made it possible to prepare thin and uniform sources containing a known aliquant of a stock solution (NAS/NRC 1962, p. 127).

For very thin sources, it is necessary either to swing the collector plate away or have it covered during initial heating in order to burn off impurities at low temperatures without volatilizing them onto the source mount. Separation from contaminants can be accomplished at the time of source preparation by considering differences in vapor pressure and carefully controlling the temperature (Coomber 1975, p. 306). The temperature at which a radionuclide will volatilize depends on the compound in which it exists, e.g., as a hydride, oxide, or halide. Sources have been prepared by thermal volatilization/sublimation for radioisotopes of manganese, chromium, cobalt, rhodium, arsenic, silver, ruthenium, technetium, and many others (Blanchard et al., 1957, p. 9; Coomber 1975, pp. 306-308). See Section 13.5, Volatilization and Distillation, for further discussion of this topic with examples.

A technique called vacuum evaporation has been used to prepare thin, uniform radioactive sources (Van der Eijk, 1973). Radioactive substances are volatilized by heating a solution in an oven under reduced pressure. Yields, usually rather low, can be improved by using a collimating oven.

16.7.5 Preparing Sources to Measure Radioactive Gases

Gaseous radionuclides most often measured include tritium, both as a vapor (^{3}HOH) and in the elemental form (^{3}H-H), ^{14}C, as CO_2, and the noble gases, ^{37}Ar, ^{41}Ar, ^{85}Kr, ^{131m}Xe, and ^{133}Xe.

Tritiated water vapor is often collected by condensation from a known volume of air (EPA 1984b). The air is drawn first through a filter to remove all particulates and then through a cold trap submerged in a dry ice/alcohol bath. A measured aliquant of the water collected is analyzed by liquid scintillation spectrometry (EPA, 1984b). Tritiated water vapor is sometimes collected by pulling air through a trap containing silica gel (SC&A, 1994). After collection, the water is distilled from the silica gel, collected, and counted in a liquid scintillation spectrometer.

Gaseous products of oxidation or combustion can be trapped in a suitable media, such as water for ^{3}H, ethanolamine for ^{14}C, peroxide for ^{35}S, and then analyzed by liquid scintillation spectrometry (NCRP, 1978, p. 211). For this method, it is very important to de-aerate the liquid prior to introducing the gas, and the temperature must be carefully controlled since gas solubilities are temperature dependent (NCRP, 1978, p. 210), generally inversely proportional to the temperature.

Although not as common nor convenient as liquid scintillation spectrometry, a gaseous radionuclide can be measured in an internal proportional counter as a component of the counter-filling gaseous mixture, usually argon, methane, or an argon-methane mixture (Friedlander and

Kennedy 1955, p. 274; NAS/NRC 1962, p. 128; Bleuler and Goldsmith 1952). For example, tritiated water can be reduced to hydrogen gas ($^3H_2$) by passing water vapor over a bed of hot zinc, and sodium carbonate can be converted to carbon dioxide ($^{14}CO_2$) by the action of an acid (NCRP, 1978, p. 211). These gases then can be mixed with a counting gas and introduced into the proportional-counter chamber. The major disadvantage of this technique is that it requires a gas handling system.

Concentrations of radioactive noble gases in the effluents of some nuclear facilities are sufficiently high that source preparation simply involves filling an evacuated vessel with the gaseous sample or flushing the vessel sufficiently to insure a 100 percent exchange (EPA, 1984b, pp. 19-20). The counting geometries (efficiencies) of the collection vessels can be determined, allowing the collected test sources to be measured directly in the vessels by gamma-ray spectrometry.

For environmental samples collected downwind of a nuclear facility, concentrating the nuclides in the gaseous sample is nearly always required prior to measurement. One example is a system, called the "Penn State Noble Gas Monitor," which was designed to measure low concentrations of radioactive noble gases (Jabs and Jester, 1976; Jester and Hepburn, 1977). Samples of environmental air are compressed in SCUBA (high pressure) bottles to 3,000 psig, providing a sample volume of 2.3 m^3. The inlet air to the compressor passes through a scrubbing train that contains particulate filters and activated charcoal to remove radioiodine. The noble-gas measurement system consists of a spherical 14.69 L, high-pressure, stainless steel vessel with a reentrant well in its base to permit insertion of a Ge detector connected to a spectrometry system. The vessel is surrounded with 2 inches of lead shielding.

There may be occasions when radioiodine is discharged into the atmosphere in several chemical forms. A molecular species filtering system, described by EPA (1990), collects four primary species of iodine on separate cartridges so that they can be measured individually. Air is pulled first through a particulate filter and then through the cartridges placed in series. The normal order of the four cartridges in the filtering system is as follows: (1) cadmium iodide media (CdI_2) for I_2 retention, (2) 4-iodophenol ($I \cdot C_6H_4 \cdot OH$) on alumina for HOI retention, (3) silver-salt (AgX) loaded zeolite or impregnated charcoal for organic iodine retention, and (4) charcoal for a breakthrough monitor. Air, at a calibrated flow, is passed through the system at a rate of one to two cubic feet per minute (cfm). When the sample-collection period is complete, the cartridges are separated, and the activities of each are measured separately by direct counting of the individual cartridges using gamma-ray spectrometry.

16.7.6 Preparing Air Filters for Counting

Air filters containing particulates may be counted directly by a proportional or scintillation detector. Minimal source preparation is normally required for directly counted filters. Some project plans may require that the mass of the particulates on filters be determined. If so required, the filters are weighed on receipt and the net particulate mass calculated by subtracting the mass of an average filter mass or, if pre-weighed, the beginning filter mass.

Actual preparation may be limited to a reduction of the size of the filter and placing it in the appropriate counting container, e.g., a planchet. If the filter is of the correct size and shape to fit directly in a counting container, no preparation may be required. Since particulate matter is deposited on the surface of the filter medium, care must be exercised in handling, particularly during size reduction, so that particulate material is not removed.

Because potentially contaminated material is relatively easily removed from a filter surface, caution is necessary to avoid contamination of detectors. If a filter is to be gamma counted it can remain in the envelope or plastic bag in which it is received for counting. The filter may be placed in such an enclosure if not received in that manner. The size of the filter may be reduced by simply folding the filter to a standard size for gamma counting.

When specific alpha- and beta-emitting nuclide analyses are required (e.g., Pu, U, Th, Am, Sr), the filter media along with the particulate material are usually ashed or dissolved and processed as any digestate by the procedure used in the laboratory.

16.7.7 Preparing Swipes/Smears for Counting

Swipes are collected to determine the level of removable surface contamination. They are normally taken on a filter paper or fabric pad by rubbing it over a predetermined surface area, nominally 100 cm^2. Swipes are routinely counted directly in a proportional counter for alpha and beta activity determination. The size of the swipe is selected to allow it to be placed in a standard-size planchet for counting. If elevated beta radioactivity is identified, a swipe may be gamma counted to determine the contributing radionuclide. Elevated alpha activity may require isotopic analyses for identification.

The precaution relative to detector contamination given above for air filters applies to swipes. All swipes should be treated as if they are contaminated until proven otherwise. In some cases swipes may be wetted with water or alcohol prior to collection of the sample. Wet swipes shall be

allowed to air dry prior to counting in order to avoid the reduction of particles reaching the detector due to absorption in the liquid remaining on the swipe.

16.8 References

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