

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.

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

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

48 **16.2 Instrument Calibration**

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

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

65 **16.2.1 Standards**

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

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

76 **16.2.2 Correspondence**

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

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

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

95 **16.2.3 Homogeneity**

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

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

104 **16.2.4 Uncertainty**

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

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

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

121 **16.3 General Test Source Characteristics**

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

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

126 **16.3.1 Geometrical Arrangement**

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

135 **16.3.2 Uniformity of Test Source Material**

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

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

154 **16.3.3 Self-Absorption and Scattering**

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

163 Because of the much smaller mass of beta particles, scattering is more pronounced in sources
164 emitting beta particles than in those emitting alpha particles. Depending on counter geometry,
165 measured beta activity can first increase as the source thickness increases, because of the
166 scattering of electrons out of the source plane and into the detector (Friedlander and Kennedy,
167 1955, pp. 276-278). At greater thicknesses, self-absorption begins to predominate, and the
168 activity eventually approaches a constant value. When this occurs, the source is said to be
169 “infinitely thick.” Counting a source at infinite thickness refers to a measurement made with a
170 source thickness such that further increasing the amount of material added would have no effect
171 on the count rate. The minimum source thickness required for this type of measurement clearly is
172 not more than the maximum range R of the particle in the source material, and is often estimated
173 to be $0.75R$ (Friedlander and Kennedy, 1955, p. 278).

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

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

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

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

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

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

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

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

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

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

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

222 **16.3.4 Counting Planchets**

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

- 227 • *Chemical reactivity.* The metal planchet must be inert to the chemicals in the test source, as
228 corrosion of the planchet surface radically alters test source absorption and geometry
229 characteristics.
- 230 • *Radioactivity.* The metal comprising the planchet should contain minimal radioactivity and,
231 although this is generally not a serious problem, the planchet background shall be measured.
- 232 • *Size.* Two-inch planchets (assuming the detector is at least that large) are often preferred for
233 gross alpha/beta counting to expedite and simplify the evaporation of liquid samples and
234 provide a greater surface area for solid samples, while 1-inch planchets are generally used for
235 alpha spectrometry test samples.
- 236 • *Cost.* Platinum planchets should not be used if stainless-steel ones are adequate for the
237 purpose.

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

243 **16.4 Test Source Preparation and Calibration for Alpha Measurements**

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

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

249 **16.4.1 Proportional Counters**

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

254 16.4.1.1 Alpha Test Source Preparation

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

260 The absorption of alpha particles in the source material (self-absorption) is quite important when
261 using proportional counters, or other ionization counters, and must be addressed when preparing
262 a test source for counting. Self-absorption is primarily a function of source thickness (t_s) and the
263 range (R_s) of the alpha particles in the source material. For a uniformly thick source, the fraction
264 of alpha particles absorbed by the source increases proportionately to $t_s/2R_s$, when $t_s < R_s$ (NCRP,
265 1978, pp.104-105). Thus, to approach absolute counting in either 2π or 4π counting geometries,
266 test sources should be prepared as thinly and uniformly as possible.

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

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

277 16.4.1.2 Proportional Counter Calibration — Alpha

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

282 **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

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

293
$$\epsilon = \frac{cps}{dps}$$

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

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

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

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

328 **16.4.2 ZnS(Ag) Scintillation Counter**

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

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

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

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

345 **16.4.3 Alpha Spectrometry With Semiconductor Detectors**

346 Semiconductor detectors for alpha particle counting are discussed in Section 15.2.2.5. Alpha-
347 energy spectra of very high resolution are attainable with semiconductor detectors if the prepared
348 test source is essentially weightless, $\leq 1 \mu\text{g}/\text{mm}^2$ (Herpers, 1986, pp. 143-145). As the thickness
349 of the test source increases, the spectral energy is degraded due to self-absorption, which
350 broadens the peak and forms a “tail” on the lower-energy side (Chapter 17). The alpha-energy
351 spectral degradation will increase, as the source thickness increases, raising the possibility of
352 overlapping peaks with a loss of spectrum integrity. Thus, it is of utmost importance to prepare
353 very thin and uniform alpha test sources for spectrometry. This may be accomplished by
354 electrodeposition or coprecipitation (ASTM, D3084), if reagents are controlled so that only small
355 (milligram) quantities of precipitate are recovered (Sections 16.6.1 and 16.7.2). For example, in
356 the coprecipitation of actinide test sources for spectral analysis, source thicknesses of 0.4 to 1
357 $\mu\text{g}/\text{mm}^2$ (0.04-0.1 mg/cm^2) are routinely achieved, which is quite adequate for producing well-
358 defined alpha spectral peaks (EPA, 1984a).

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

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

374 **16.4.4 Liquid-Scintillation Spectrometer**

375 With proper scintillators, liquid scintillation can be used to measure alpha-particle emitters
376 (Passo and Cook, 1994) (Section 15.2.2.4). Although the relatively high background of liquid
377 scintillation counting restricts the sensitivity relative to other counting techniques, e.g., internal
378 proportional counting or the use of ZnS(Ag) screens, the ease of source preparation and the
379 nearly 100 percent counting efficiency are advantages often exploited (Hemingway, 1975, p.
380 146). The separation of alpha- and beta-particle counts attained in the spectrometer can be
381 enhanced by proper scintillator choice. Ultima Gold AB™ was designed specifically to maximize
382 alpha/beta separation in aqueous solutions and, in other studies, poor alpha/beta separation has
383 been overcome by making the standard cocktail 20 percent in naphthalene (Passo and Cook,
384 1994, pp. 3-11 to 3-12). It is believed that naphthalene improves the alpha/beta separation by
385 acting as an intermediate in the energy transfer process between the solvent and the fluor
386 (McDowell, 1986).

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

395 Some actinides (U and Th) and transuranics (Np, Pu, Am, and Cm) have been measured by a
396 procedure that involves "Extraction Scintillation Techniques" (Passo and Cook, 1994, pp. 6-1 to
397 6-2 and 13-1 to 13-6). An extraction agent, e.g., bis(2-ethylhexyl) phosphoric acid (HDEHP), is
398 mixed either with a toluene or a di-isopropylnaphthalene (DIN) based cocktail. The alpha
399 emitter, in the aqueous laboratory sample, is extracted into the scintillation mixture and counted
400 by liquid scintillation. The discussion in Section 16.5.2.1 can be applied to both alpha and beta
401 particles.

402 **16.5 Characteristics of Sources for Beta Measurements**

403 **16.5.1 Proportional Counters**

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

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

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

419 16.5.1.1 Beta Test Source Preparation

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

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

431 16.5.1.2 Proportional Counter Calibration — Beta

432 As in other calibrations, proportional counters used for beta-particle analysis shall be calibrated
433 with NIST traceable standards in a manner that is totally consistent with the counting of test
434 sources. When possible, the radionuclide to be quantified should be used as the calibration
435 source. For gross beta analysis, the radionuclides presented in Table 16.2 have been
436 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., ^3H , ^{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 ^1H and ^3H (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

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

473 16.5.2.1 Liquid Scintillation Test Source Preparation

474 The preparation of a laboratory sample for a liquid-scintillation spectrometer usually is relatively
475 simple and fast. The radionuclide to be measured is isolated in a solution, which is then
476 introduced into and thoroughly mixed with one of a variety of ready-to-use commercially
477 available liquid scintillators. This mixture is often referred to as a scintillation “cocktail.” The
478 liquid scintillator is an emulsion system, usually consisting of an aromatic solvent containing the
479 appropriate scintillator mixed with a detergent (NCRP, 1978, pp.168-169). If a sample is
480 insoluble in the scintillator, it can be ground to a fine powder, stirred into the scintillator until a
481 homogeneous mixture is formed, and solidified with a gelling agent (Friedlander et al., 1981, p.
482 303).

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

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

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

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

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

514 16.5.2.1 Liquid-Scintillation Spectrometer Calibration

515 When the quenching of a group of test sources is predictable, e.g., distilled drinking water (EPA,
516 1980; ASTM D4107), a counting efficiency is determined for the group by placing a known
517 quantity of reference material in the source medium and scintillation solution under identical
518 conditions (vials and volumes) as the sample medium.

519 Except for test sources with very predictable amounts of quenching, it may be necessary to
520 determine a counting efficiency for each laboratory sample. Two methods of determining
521 counting efficiency are available: internal standardization and external standardization (NCRP,
522 1978).

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

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

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

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

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

544 **16.6 Characteristics of Sources for Gamma-Ray Measurements**

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

552 **16.6.1 Gamma Test Source Preparation**

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

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

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

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

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

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

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

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

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

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

598 **16.6.2 Gamma Spectrometer Calibration**

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

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

607 MARLAP recommends that calibration data for gamma spectroscopy calibration be obtained
608 from the National Nuclear Data Center at Brookhaven National Laboratory ([http://www.nndc.
609 bnl.gov/nndc/nudat/](http://www.nndc.bnl.gov/nndc/nudat/)). Calibration data are readily available for common radionuclides, including
610 ²¹⁰Pb, ²⁴¹Am, ¹⁰⁹Cd, ⁵⁷Co, ¹⁴¹Ce, ¹³⁹Ce, ²⁰³Hg, ⁵¹Cr, ¹¹³Sn, ⁸⁵Sr, ¹³⁷Cs, ⁵⁴Mn, ⁸⁸Y, ⁶⁵Zn, ⁶⁰Co, and ⁴⁰K.
611 For more information on gamma spectrometry calibration see ANSI 42.14. (Also see Section
612 17.3.1.6 on gamma calibration.)

613 **16.7 Methods of Test Source Preparation**

614 **16.7.1 Electrodeposition**

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

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

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

644 Electrodeposition is applicable to more than 30 radionuclides. The main advantage of
645 electrodeposited sources over those from other methods of preparation is their extremely thin,
646 uniform deposit of a radionuclide on a plate, which permits high resolution spectroscopy;
647 however, the yield is often not quantitative (Adloff and Guillaumont, 1993, p. 158). Thus, the
648 yield must be monitored with the inclusion of a known quantity of an isotope, which is deposited
649 simultaneously with the analyte. Radioactive sources of the following elements have been
650 prepared successfully by electrodeposition (DOE, 1997; Blanchard et al., 1960; Johnston et al.,
651 1991.)

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

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

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

680 Test sources of actinides are often prepared by electrodeposition with yields of 90 percent and
681 higher (DOE, 1997; EPA, 1979; Sill et al., 1974; Puphal and Olsen, 1972; Kressin, 1977; Talvite,
682 1972; Mitchell, 1960; Shinohara and Kohno, 1989, pp. 41-45). In addition, ^{54}Mn sources have
683 been successfully prepared by the electrodeposition from mixed-solvent electrolytes onto
684 stainless steel planchets (Sahoo and Kannan, 1997, pp. 185-190).

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



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

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

695 A similar technique, called internal electrolysis, is performed by selecting electrodes that have a
696 large difference in potential. A conventional electrolytic cell containing an acid solution of the
697 radionuclide to be deposited may be used. A magnesium ($E^\circ = +2.37$ volts) strip, for example, is
698 inserted into the electrolyte and connected by an external circuit to the inert metal cathode
699 (planchet), usually platinum. A spontaneous current flows and deposition on the cathode will
700 occur. The conditions at the inert cathode are exactly the same as if an external voltage were
701 applied; however, longer electrolysis times are necessary to achieve quantitative recoveries. Very
702 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
703 this technique, with greater than 96 percent recovery in all cases (Blanchard et al., 1957, pp. 46-
704 54; Van der Eijk et al., 1973).

705 **16.7.2 Coprecipitation**

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

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

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

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	<u>Radionuclide</u>	<u>Carrier</u>	<u>References</u>
726	³² P	MgNH ₄ PO ₄	a
727	⁵¹ Cr	BaCrO ₄	a
728	^{89/90} Sr	SrCO ₃	a,b,c
729	⁹⁰ Y	Y ₂ (C ₂ O ₄) ₃	a,b,c
730	¹³¹ I	PdI ₂	a,b,c
731	¹³⁷ Cs	Cs ₂ PtCl ₆	b
732	¹⁴⁷ Pm	Nd ₂ (C ₂ O ₄) ₃	a
733	²¹⁰ Bi	BiOCl	a
734	²²⁶ Ra	BaSO ₄	b
735	Th	Ce(IO ₄) ₄	d
736	Th	LaF ₃	a,b
737	U	LaF ₃ (NdF ₃)	a,b,(f)
738	Np	LaF ₃	b
739	Pu	LaF ₃ (NdF ₃)	a,b,d,(f)
740	Am	LaF ₃ (NdF ₃)	a,b,d,(f)
741	Cm	LaF ₃	b
742	Th	Ce(OH) ₂	e
743	Np	Ce(OH) ₂	e
744	Pu	Ce(OH) ₂	e
745	Am	Ce(OH) ₂	e
746	Cm	Ce(OH) ₂	e
747	U	UF ₃	e
748			
749	a EPA (1984)	c DOE (1997)	e Sill (1981)
750	b EPA (1980)	d Hindman (1983)	f Hindman (1986)

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

756 Another precipitation technique has been applied to preparing radioactive sources. Source
 757 preparation by precipitation can be conducted in a desiccator fitted with a valve to allow first the
 758 evacuation of the desiccator and then the admission of a precipitating gas, such as ammonia
 759 (NH₃) or hydrogen sulfide (H₂S) (Blanchard et al., 1957, pp. 26-31; Van der Eijk et al., 1973). A
 760 carrier is added to the sample and a know quantity is pipetted onto a planchet. The planchet
 761 containing the test source solution is placed in the desiccator and exposed to a precipitating gas
 762 for one to two hours. This period of time allows settling to occur. The test source is removed
 763 from the desiccator and evaporated beneath a heat lamp. Using an AlCl₃ carrier in an ammonia
 764 atmosphere, Yoshida et al. (1977) prepared uniformly deposited radioactive sources of ⁵⁹Fe, ⁶⁰Co,
 765 ⁹⁵Nb, ¹⁰³Ru, and ¹⁹⁸Au by this technique.

766 **16.7.3 Evaporation**

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

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

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

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

798 EPA's (1980) prescribed Method 900.0 for measuring gross alpha and beta radioactivity in
799 drinking water suggests that the sample aliquant be limited to what will produce 5 mg/cm² of
800 solids on the planchet. Thus, for a 2-inch planchet (20 cm²), an aliquant containing 100 mg of
801 non-volatile dissolved solids is the recommended maximum test source mass.

802 After a radionuclide in solution has been purified by chemical techniques, i.e., impurities
803 removed, the solution can be transferred to a planchet and evaporated to dryness, as described
804 above. Evaporation of a laboratory sample after purification is used by the EPA to measure ²²⁸Ac
805 in the analysis for ²²⁸Ra (EPA, 1984a), and sources of thorium, isolated from marine carbonates,
806 have been prepared by evaporation for measurement by alpha spectrometry (Blanchard et al.,
807 1967). Measured count rates of identified radionuclides, for which absorption curves have been
808 prepared, can be adjusted for self absorption in evaporated test sources.

809 In the case of all dry sources, steps should be taken to prevent solids from exiting the planchet,
810 which will affect the measurement and, in time, contaminate the detector. Sources consisting of
811 loose, dry material, or with a tendency to flake, should be covered with thin plastic or
812 immobilized by evaporating a few drops of a lucite-acetone solution on the solid deposit (PHS,
813 1967, p. 21).

814 **16.7.4 Thermal Volatilization/Sublimation**

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

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

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

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

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

842 **16.7.5 Preparing Sources to Measure Radioactive Gases**

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

845 Tritiated water vapor is often collected by condensation from a known volume of air (EPA
846 1984b). The air is drawn first through a filter to remove all particulates and then through a cold
847 trap submerged in a dry ice/alcohol bath. A measured aliquant of the water collected is analyzed
848 by liquid scintillation spectrometry (EPA, 1984b). Tritiated water vapor is sometimes collected
849 by pulling air through a trap containing silica gel (SC&A, 1994). After collection, the water is
850 distilled from the silica gel, collected, and counted in a liquid scintillation spectrometer.

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

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

860 Kennedy 1955, p. 274; NAS/NRC 1962, p. 128; Bleuler and Goldsmith 1952). For example,
861 tritiated water can be reduced to hydrogen gas ($^3\text{H}_2$) by passing water vapor over a bed of hot
862 zinc, and sodium carbonate can be converted to carbon dioxide ($^{14}\text{CO}_2$) by the action of an acid
863 (NCRP, 1978, p. 211). These gases then can be mixed with a counting gas and introduced into
864 the proportional-counter chamber. The major disadvantage of this technique is that it requires a
865 gas handling system.

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

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

882 There may be occasions when radioiodine is discharged into the atmosphere in several chemical
883 forms. A molecular species filtering system, described by EPA (1990), collects four primary
884 species of iodine on separate cartridges so that they can be measured individually. Air is pulled
885 first through a particulate filter and then through the cartridges placed in series. The normal order
886 of the four cartridges in the filtering system is as follows: (1) cadmium iodide media (CdI_2) for I_2
887 retention, (2) 4-iodophenol ($\text{I} \cdot \text{C}_6\text{H}_4 \cdot \text{OH}$) on alumina for HOI retention, (3) silver-salt (AgX)
888 loaded zeolite or impregnated charcoal for organic iodine retention, and (4) charcoal for a
889 breakthrough monitor. Air, at a calibrated flow, is passed through the system at a
890 rate of one to two cubic feet per minute (cfm). When the sample-collection period is complete,
891 the cartridges are separated, and the activities of each are measured separately by direct counting
892 of the individual cartridges using gamma-ray spectrometry.

893 **16.7.6 Preparing Air Filters for Counting**

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

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

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

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

912 **16.7.7 Preparing Swipes/Smears for Counting**

913 Swipes are collected to determine the level of removable surface contamination. They are
914 normally taken on a filter paper or fabric pad by rubbing it over a predetermined surface area,
915 nominally 100 cm². Swipes are routinely counted directly in a proportional counter for alpha and
916 beta activity determination. The size of the swipe is selected to allow it to be placed in a
917 standard-size planchet for counting. If elevated beta radioactivity is identified, a swipe may be
918 gamma counted to determine the contributing radionuclide. Elevated alpha activity may require
919 isotopic analyses for identification.

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

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

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