

10 FIELD AND SAMPLING ISSUES THAT AFFECT LABORATORY MEASUREMENTS

Part I: Generic Issues

10.1 Introduction

The primary purpose of this chapter is to provide guidance on issues that affect laboratory measurements to project planners and managers tasked with developing a field sampling plan. Specifically, this chapter provides guidance on activities conducted primarily after the proper collection of the sample. Sampling design and collection are beyond the scope of MARLAP. A field sampling plan should be a comprehensive document that provides detailed guidance for collecting, preparing, preserving, shipping, tracking field samples, and recording field data. The principal objective of a well-designed sampling plan is to provide representative samples of the proper size for analysis. Critical to the sampling plan are outputs of the systematic planning process, which commonly define the Analytical Protocol Specifications (APS) and the Measurement Quality Objectives (MQO) that must be met. While a comprehensive discussion that extends to field sampling strategies is beyond the scope of this chapter, specific aspects of sample collection methods and physical preparation and preservation of samples warrant further discussion because they impact the analytical process and the data quality.

This chapter is divided into two main parts. Part I identifies general elements of a field sampling plan and provides project planners with general guidance. Part II provides more detailed information. Matrix-specific guidance and technical data are presented for liquid, solid, airborne, and surface contaminants requiring field sampling. This information will assist project planners further in the development of standard operating procedures (SOPs) and training for field personnel engaged in preparation and preservation of field samples.

The need to specify sample collection methods, and preparation and preservation of field samples, is commonly dictated by one or more of the following:

- The systematic planning process that identifies the type, quality, and quantity of data needed to satisfy a decision process;
- The potential alteration of field samples by physical, chemical, and biological processes during the time between collection and analysis;
- Requirements specified by the analytical laboratory pertaining to sample analysis;
- Requirements of analytical methods; and
- Requirements of regulators (e.g., Department of Transportation).

33 **10.1.1 The Need for Establishing Channels of Communication**

34 Of critical importance to the effective design of a sampling plan are the input and recommen-
35 dations of members representing: (1) the field sampling team; (2) the health physics professional
36 staff; (3) the analytical laboratory; (4) statistical and data analyses; (5) quality assurance
37 personnel, and (6) end-users of data.

38 Beyond the initial input that assist the project planners in the design of the sampling plan, it is
39 equally important to maintain open channels of communication among key members of the
40 project team throughout the process. For example, the analytical laboratory should be provided
41 with contacts from the field sampling team to ensure that modifications discrepancies and
42 changes are addressed and the timely resolution of potential problems.

43 Communication among project staff, field personnel, and the laboratory offer a means to
44 coordinate activities, schedules, and sample receipt. Project planning documents generated from
45 the systematic planning process, such as APS and statements of work (SOWs), should be
46 consulted, but they cannot address all details. Additional communication likely will be necessary.
47 Communication conveys information about the number and type of samples the laboratory can
48 expect at a certain time. Documentation with special instructions regarding the samples should be
49 received before the samples arrive. This information notifies the laboratory of any health and
50 safety concerns so that laboratory personnel can implement proper contamination management
51 practices. Health and safety concerns may affect analytical procedures, sample disposition, etc.
52 The analytical laboratory should have an initial understanding about the relative number of
53 samples that will be received and the types of analyses that are expected for specific samples.
54 Furthermore, advance communications allow laboratory staff to adjust to modifications,
55 discrepancies, and changes.

56 **10.1.2 Developing Field Documentation**

57 The field organization must conduct its operations in such a manner as to provide reliable
58 information that meets the data quality objectives (DQOs). To achieve this goal, all relevant
59 procedures pertaining to sample collection and processing should be based on documented
60 standard operating procedures that include the following activities:

- 61 • Developing a technical basis for defining the size of individual samples;
- 62 • Selecting field equipment and instrumentation;
- 63 • Using proper sample containers and preservatives;
- 64 • Using consistent container labels and sample identification codes;
- 65 • Documenting field sample conditions and exceptions;
- 66 • Documenting sample location;
- 67 • Tracking, accountability and custody, and shipment forms;

- 68 • Legal accountability, such as chain-of-custody record, when required;
- 69 • Selecting samples for field QC program;
- 70 • Decontaminating equipment and avoiding sample cross-contamination;
- 71 • Sample packaging, shipping, and tracking; and
- 72 • Health and safety plan.

73 **10.2 Field Sampling Plan: Non Matrix Specific Issues**

74 **10.2.1 Determination of Analytical Sample Size**

75 When collecting environmental samples for radioanalysis, an important parameter for field
76 personnel is the mass, volume, or weight of an individual sample that must be collected. The
77 required minimum sample size is best determined through the collective input of project
78 planners, field technicians, and laboratory personnel who must consider the likely range of the
79 contaminant concentrations, the type of radiation emitted by constituents or analytes (α , β , γ),
80 field logistics, and the radioanalytical methods that are to be employed. For samples to yield
81 useful data, it is important to have a quantitative understanding of the relationship between
82 sample size and project specific requirements.

83 **10.2.2 Field Equipment and Supply Needs**

84 Before starting field sampling activities, all necessary equipment and supplies should be
85 identified, checked for proper operation and availability, and—when appropriate—pre-
86 assembled. Instrumentation and equipment needs will depend not only on the medium to be
87 sampled, but also on the accessibility of the medium and the physical and chemical properties of
88 radionuclide contaminants under investigation.

89 Independent of specialized field equipment and instrumentation, field sampling supplies
90 commonly include the following:

- 91 • Sampling devices (e.g., trowel, hand auger, soil core sampler, submersible water pump, high
92 volume air filter, etc.);
- 93 • Sampling preparation equipment (e.g., weighing scales, volume measuring devices, soil
94 screening sieves, water filtering equipment, etc.);
- 95 • Sample preservation equipment and agents (e.g., refrigeration, ice, formaldehyde or acid
96 additives);
- 97 • Personnel protective gear (e.g., respiratory protective devices, protective clothing such as
98 gloves and booties, life-preservers, etc.);

- 99 • Proper writing utensils (e.g., permanent pens and markers);
- 100 • Field logbooks and field tracking forms;
- 101 • Maps, distance measuring equipment, global positioning systems, or other location-
102 determining equipment;
- 103 • Field sampling flags or paint;
- 104 • Chain-of-custody (COC) forms;
- 105 • Sample tags, labels, documents;
- 106 • Appropriately labeled sample containers;
- 107 • Shipment containers and packing materials that meet DOT regulations;
- 108 • Shipment forms;
- 109 • Analysis request form identifying the type of radioanalysis to be performed; and
- 110 • Health and Safety Plan requirements (medical kit, etc.).

111 **10.2.3 Selection of Sample Containers**

112 There are several physical and chemical characteristics that must be considered when selecting a
113 suitable container for shipping and storing samples. Important characteristics include the
114 container material and its size, configuration, and method for ensuring a proper seal.

115 10.2.3.1 Container Material

116 Sample containers must provide reasonable assurance of maintaining physical integrity (i.e.,
117 against breakage, rupture, or leakage) during handling, transport, and potentially long periods of
118 storage. The most important factor to consider in container selection is the chemical
119 compatibility between container material and sample. Containers may include ordinary bottle
120 glass, borosilicate glass (such as Pyrex or Corex), plastics (e.g., high density polyethylene—
121 HDPE), low density polyethylene, polycarbonate, polyvinyl chloride (PVC), fluorinated ethylene
122 propylene (Reflon), or polymethelpentene. For select samples, the choice of containers may
123 require metal construction or be limited to paper envelopes.

124 10.2.3.2 Container Opening and Closure

125 Selection of a suitable container also must consider the ease with which the sample is introduced
126 into the container. For example, a wide-mouthed container will provide easier access for the
127 introduction and withdrawal of sample material and eliminate spills or the need for additional
128 tools or equipment (e.g., funnel) that may become a source of cross contamination among
129 samples.

130 Equally important is the container closure or seal. As a rule, snap-on caps should not be
131 considered for liquid samples because they do not ensure a proper seal. Even when screw caps
132 are used, it is frequently prudent to protect against vibration by securing the cap with electrical or
133 duct tape. A proper seal is important for air samples, such as radon samples. The container cap
134 material, if different from the container material, must be equally inert with regard to sample
135 constituents.

136 10.2.3.3 Sealing Containers

137 Tamper-proof seals offer an additional measure to ensure sample integrity. A simple example
138 includes placing a narrow strip of paper over a bottle cover and then affixing this to the container
139 with a wide strip of clear tape (EPA, 1987, Exhibit 5-6, example of custody seals). The paper
140 strip can be initialed and dated in the field to indicate the staff member who sealed the sample
141 and the date of the seal. Individually sealing each sample with a custody seal with the collector's
142 initials and the date the sample was sealed may be required by the project. The seal ensures legal
143 defensibility and integrity of the sample at collection. Tamper-proof seals should only be applied
144 once field processing and preservation steps are completed. Reopening this type of sealed
145 container in the field might warrant using a new container or collecting another sample.

146 10.2.3.4 Precleaned and Extra Containers

147 The reuse of sample containers is discouraged because traces of radionuclides might persist from
148 initial container use to subsequent use. The use of new containers for each collection removes
149 doubts concerning radionuclides from previous sampling. New containers might also require
150 cleaning (ASTM D5245) to remove plasticizer used in container production or to pretreat glass
151 surfaces. Retaining extra empty containers from a new lot or a special batch of precleaned and
152 treated containers offers the laboratory container blanks for use as part of quality control. Extra
153 containers are also useful for taking additional samples as needed during field collection and to
154 replace broken or leaking containers.

155 **10.2.4 Container Label and Sample Identification Code**

156 Each sample can only be identified over the life of a study if a form of *permanent identification*
157 is provided with or affixed to the container or available in sample log. The most useful form of

158 identification utilizes a *unique identifier* for each sample. Such unique identification codes
159 ensure the project's ability to track individual samples. The standard operating procedure (SOP)
160 that addresses sample identification should describe the method to be used to assure that samples
161 are properly identified and controlled in a consistent manner. Containers sometimes may be pre-
162 labeled with identification numbers already in place.

163 Any identification recorded on a container or a label affixed to the container should remain with
164 the container throughout sample processing and storage. The identification information should be
165 written with a permanent marker—especially if the labels are exposed to liquids. Information can
166 be recorded directly on the container or on plastic or paper tags securely fixed to the container.
167 However, tags are more likely to become separated from containers than are properly secured
168 labels.

169 Labels, tags, and bar codes should be rugged enough so no information is lost or compromised
170 during field work, sample transport, or laboratory processing. Transparent tape can be used to
171 cover the label once it is completed. The tape protects the label, adds moisture resistance,
172 prevents tampering with the sample information, and helps secure the label to the container.

173 The project manager needs to determine if a sample number scheme may introduce bias into the
174 analysis process. That is, the lab may be aware of trends or locations from the sample
175 identification and this could influence their judgment as to the anticipated result and thereby
176 introduce actions on the part of lab personnel that they would not otherwise take. The project
177 manager needs to determine the applicability of electronic field data recorders and the issue of
178 electronic signatures for the project.

179 A unique identifier can include a code for a site, the sample location at the site, and a series of
180 digits identifying the year and day of year (e.g., "1997-127" uses the Julian date, and "062296"
181 describes a month, day, and year). Alternatively, a series of digits can be assigned sequentially by
182 site, date, and laboratory destination. The use of compass headings and grid locations also
183 provides additional unique information (e.g., "NW fence, sampled at grid points: A1 through
184 C25, 072196, soil"). With this approach, samples arriving at a laboratory are then unique in two
185 ways. First, each sample can be discriminated from materials collected at other sites. Second, if
186 repeat samples are made at a single site, then subsequent samples from the same location are
187 unique only by date. Labeling of samples sequentially might not be appropriate for all studies.
188 Bar coding may reduce transcription errors and should be evaluated for a specific project.

189 **10.2.5 Field Data Documentation**

190 All information pertinent to field sampling is documented in a log book or on a data form. The
191 log book should be bound and the pages numbered consecutively and forms should be page-
192 numbered and dated. Where the same information is requested routinely, preprinted log books or
193 data sheets will minimize the effort and will standardize the presentation of data. Even when

194 standardized preprinted forms are used, all information recorded should be in indelible ink, with
195 all entry errors crossed out with a single line and initialed. The color of ink used should be
196 compatible with the need to copy that information. All entries should be dated and signed on the
197 date of entry. Initials should be legible and traceable, so that it is clear who made the entry.

198 Whenever appropriate, log or data form entries should contain—but are not limited to—the
199 following:

- 200 • Identification of Project Plan or Sampling Plan;
- 201 • Location of sampling (e.g., reference to grid location, maps, photographs, location in a
202 room);
- 203 • Date and time of sample collection;
- 204 • Sample medium (e.g., surface water, soil, sediment, sludge, etc.);
- 205 • Suspected radionuclide constituents;
- 206 • Sample-specific ID number;
- 207 • Sample volume, weight, depth;
- 208 • Sample type (e.g., grab, composite);
- 209 • Sample preparation used (e.g., removal of extraneous matter);
- 210 • Sample preservation used;
- 211 • Requested analyses to be performed (e.g., gross beta/gamma, gamma spectroscopy for a
212 specific radionuclide, radiochemical analysis);
- 213 • Sample destination including name and address of analytical laboratory;
- 214 • Names of field persons responsible for collecting sample;
- 215 • Physical and meteorological conditions at time of sample collection;
- 216 • Special handling or safety precautions;

- 217 • Recommendations regarding time to date of analysis that reflect (1) the loss of radioactivity
218 due to natural decay, (2) the ingrowth and secular equilibrium of short-lived progeny, or (3)
219 the potential loss of radioactivity due to evaporation or volatility; and
- 220 • Signatures or initials of appropriate field personnel. When using initials, ensure that they can
221 be uniquely identified with an individual.

222 Labels affixed to individual sample containers should contain key information that is an abstract
223 of log book data sheets. When this is not practical, a copy of individual sample data sheets may
224 be included along with the appropriately ID-labeled sample.

225 **10.2.6 Field Tracking, Custody, and Shipment Forms**

226 A sample tracking procedure must be in place for all projects in order that the proper location and
227 identification of samples is maintained throughout the process from collection through handling,
228 preservation, storage, transfer to laboratory, and disposal. The term “tracking,” when used here,
229 connotes a tracking and accountability process that meets generally acceptable laboratory
230 practices as described by accrediting bodies, but is less stringent than a formal chain-of-custody
231 process. Tracking also develops a record of all individuals responsible for the custody and
232 transfer of the samples. Chapter 4 (*Project Plan Documents*) discusses the process of tracking
233 and accountability. Also, Chapter 11 (*Sample Receipt, Inspection, and Tracking*) discusses the
234 laboratory process of tracking.

235 When transferring the possession of samples, the individuals relinquishing and the individuals
236 receiving the samples should sign, date, and note the time on the form. A standardized form
237 should be designed for recording tracking or formal chain-of-custody information related to
238 tracking sample possession. If samples are to be split and distributed to more than one analytical
239 laboratory, multiple forms will be needed to accompany sample sets. The sample collector is
240 responsible for initiating the sample tracking record. The following information is considered
241 minimal for sample tracking:

- 242 • Name of project;
243 • Sampler’s signature;
244 • Sample ID;
245 • Sample location
246 • Date and time sampled;
247 • Sample type;
248 • Preservatives;
249 • Number of containers;
250 • Analysis required;
251 • Signatures of persons relinquishing, receiving, and transporting the samples;
252 • Signature for laboratory receipt;

- 253 • Method of shipment or carrier and air bill when shipped or shipping manifest identification
254 upon receipt; and
- 255 • Comments regarding the integrity of shipping container and individual samples.

256 **10.2.7 Chain of Custody**

257 The legal portion of the tracking and handling process that ensures legal defensibility from
258 sample collection to data reporting has become relatively standardized and is referred to as the
259 chain-of-custody (COC) process (APHA, 1996). Guidance is provided in “Standard Practice for
260 Sampling Chain-of-Custody Procedures” (ASTM D4840) and NIOSH (1983). The level of
261 security required to maintain an adequate chain of custody is that necessary to establish a
262 “reasonable probability” that the sample has not been tampered with. For court proceedings, the
263 requirements are established in law. COC procedures are important in demonstrating sample
264 control when litigation is involved. In many cases, Federal, State or local agencies may require
265 that COC be maintained for specific projects. COC is usually not required for samples that are
266 generated and immediately tested within a facility or continuous (rather than discrete or
267 integrated) samples that are subject to real- or near-real-time analysis (e.g., continuous
268 screening).

269 When COC is required, the custody information is recorded on a COC form. Chain-of-custody
270 documents vary by organization. Communication between field and laboratory personnel is
271 critical to the successful use of COC. Any error made on a custody form is crossed out with a
272 single line and dated and initialed. Use of correction ink or obliteration of data is not acceptable.
273 Inform the laboratory when COC is required before the samples are received (see Section 11.2
274 for further information). The COC documents are signed by personnel who collect the samples.
275 A chain-of-custody record accompanies the shipment and one or more copies are distributed to
276 the project coordinator or other office(s) where field and laboratory records are maintained. An
277 example of a COC form is shown in Figure 10.1. Additional information and examples of
278 custody forms are illustrated by EPA (1987) and EPA (1994).

279 **10.2.8 Field Quality Control**

280
281 A project plan should have been developed to ensure that all data are accurate and that decisions
282 based on these data are technically sound and defensible. The implementation of a project plan
283 requires quality control (QC) procedures. QC procedures, therefore, represent specific tools for
284 measuring the degree to which quality assurance objectives are met. Field quality control
285 measures are comprehensively discussed in ASTM D5283.

286 While some types of quality control (QC) samples are used to assess analytical process, field
287 quality control samples are used to assess the actual sampling process. The type and frequency of
288 these field QC samples must be specified by the project planning process along with being
289 included in the project planning documents and identified in the sampling plan. Definitions for

CHAIN-OF-CUSTODY RECORD									
FIELD IDENTIFICATION NUMBER	FIELD LOCATION	DATE	TIME	SAMPLERS (Signature)					
				SAMPLE MATRIX			SEQ. No.	No. of Containers	Analysis Required
				Water	Soil	Air			
Relinquished by: (Signature)				Received by: (Signature)				Date/Time /	
Relinquished by: (Signature)				Relinquished by: (Signature)				Date/Time /	
Relinquished by: (Signature)				Received by: (Signature)				Date/Time /	
Received by: (Signature)				Received by Laboratory for field analysis: (Signature)				Date/Time /	
Dispatched by: (Signature)			Date	Time	Received for Laboratory by:			Date/Time /	
Method of Shipment:									
Distribution: Orig. - Accompany Shipment 1 Copy – Survey Coordinator Field Files									

FIGURE 10.1—Example of chain-of-custody record.

290 certain types of field QC samples can be found in ASTM D5283 and MARSSIM (2000).

291 **10.2.9 Decontamination of Field Equipment**

292 Sampling SOPs must describe the recommended procedure for cleaning field equipment before
 293 and during the sample collection process, as well as any pretreatment of sample containers. The
 294 SOPs should include the cleaning materials and solvents used, the purity of rinsing solution or
 295 water, the order of washing and rinsing, associated personnel safety precautions, and the disposal
 296 of cleaning agents.

297 Detailed step-by-step procedures for the decontamination of field equipment used in the
298 sampling of low-activity soils, soil gas, sludges, surface water, and ground water are given in
299 ASTM D5608.

300 **10.2.10 Packing and Shipping**

301 The final responsibility of field sampling personnel is to properly prepare and package samples
302 for transport or shipment by a commercial carrier. All applicable State and Federal shipping
303 requirements, as discussed later in this section, must be followed. Samples transported over
304 shorter distances by the sampling or testing agency by way of automobile, van, or truck will
305 require less stringent packing requirements. In most instances, placing sealed sample containers
306 within cardboard boxes (or similar containers) in which individual samples are sufficiently
307 cushioned to guard against bumping, rolling, or dropping, is adequate.

308 When samples must be shipped by way of a commercial carrier or the U.S. Postal Service,
309 containers must be designed to protect samples against crushing forces, impacts, and severe
310 temperature fluctuations. Within each shipping container, the cushioning material (sawdust,
311 rubber, polystyrene, urethane foam, or material with similar resiliency) should encase each
312 sample completely. The cushioning between the samples and walls of the shipping containers
313 should have a minimum thickness of one inch. A minimum thickness of two inches should be
314 provided on the container floor.

315 Consideration must also be given to protect samples against potentially adverse impacts of
316 temperature fluctuations. When appropriate, sample protection against freezing, thawing,
317 sublimation, evaporation, or extreme temperature variation may require that the entire interior
318 surface of the shipping container be lined with an adequate layer of insulation. In many instances,
319 the insulating material may also serve as the cushioning material.

320 When metal containers are used, the requirements for container security, cushioning, and insula-
321 tion apply equally. For smaller volume and low-weight samples, properly lined containers
322 constructed with laminated fiberboard, plastic, or reinforced cardboard outer walls also may be
323 used.

324 When samples are shipped as liquids in glass or other breakable sample containers, additional
325 packaging precautions may have to be taken. Additional protection is obtained when sample
326 containers are shipped in nested containers, in which several smaller containers (i.e., inner
327 containers) are packed inside a second larger container (i.e., the outer pack or overpack). To
328 contain any spills of sample material within the shipping container, it is advisable either to wrap
329 individual samples or to line the shipping container with absorbent material, such as asbestos-
330 free vermiculite or perlite.

331 For proper packaging of liquid samples, additional guidance has been given by EPA (1987) and
332 includes the following:

- 333 • All sample bottles are taped closed;
- 334 • Each sample bottle is placed in a plastic bag and the bag is sealed;
- 335 • Each sample bottle may be placed in a separate metal can filled with vermiculite or other
336 packing material, then the lid may be fixed to the can with tape;
- 337 • The cans are placed upright in a cooler that has its drain plug taped closed, inside and out,
338 and lined with a plastic bag; and
- 339 • The cooler is filled with packing material—“bubble wrap” or cardboard separators may be
340 used—and closed with sealing tape.

341 Field screening measurements are made for compliance with Department of Transportation
342 regulations, 49 CFR Parts 170 through 189, as well as compliance with the laboratory’s U.S.
343 NRC (10 CFR Part 71) and Agreement State license. International requirements may also apply.
344 See International Air Transport Association (IATA) Dangerous Goods Regulations for additional
345 guidance. These regulations not only set contamination and dose limits for shipping containers,
346 but also describe the types of containers and associated materials that are to be used based on the
347 total activity and quantity of materials shipped. When the samples are screened in the field with
348 survey instrumentation, the results should be provided to the laboratory. This information should
349 also state the distance used from the probe to the packing container wall. Measurements normally
350 are made in contact or at one meter. The readings in contact are most appropriate for laboratory
351 use. The screening measurements in the field are mainly for compliance with transportation
352 requirements and are usually in units of exposure. Laboratory license requirements are usually by
353 isotope and activity. Project planning and communication are essential to ensure that a specific
354 set of samples can be transported, received, and analyzed safely while complying with applicable
355 rules and regulations.

356
357 The external surface of each shipping container must be labeled clearly, contain information
358 regarding the sender and receiver, and should include the respective name and telephone number
359 of a contact. When required, proper handling instructions and precautions should be clearly
360 marked on shipping containers. Copies of instructions, shipping manifest or container inventory,
361 chain of custody, and any other paperwork that is enclosed within a shipping container should be
362 safeguarded by placing documents within a sealed protected envelope.

363 **10.2.11 Worker Health and Safety Plan**

364 In some cases, field samples will be collected where hazardous agents or site conditions might
365 pose health and safety considerations for field personnel. These can include chemical, biological,
366 and radiological agents, as well as common industrial hazards associated with machinery, noise
367 levels, and heat stress. The health and safety plan established in the planning process should be

368 followed. For the Department of Defense (DOD), these plans may include imminent threats to
369 life, such as unexploded ordnance, land mines, hostile forces, chemical agents, etc.

370 10.2.11.1 Physical Hazards

371 MECHANICAL EQUIPMENT

372 Personnel working with hand-held tools (e.g., sledge hammers used for near-surface coring) or
373 power tools and equipment are subject to a variety of hazards. For example, personnel drilling
374 monitoring wells are exposed to a variety of potential mechanical hazards, including moving
375 machinery, high-pressure lines (e.g., hydraulic lines), falling objects, drilling through under-
376 ground utilities, flying machinery parts, and unsafe walking and working surfaces. The
377 consequences of accidents involving these physical hazards can range from minor to fatal injury.

378 At a minimum, workers should be required to wear protective clothing, which includes hard hats,
379 gloves, safety glasses, coveralls (as an option) and steel-toed safety shoes. Workers required to
380 climb (e.g., ladders, drilling masts) must be required to wear harnesses and lanyards and be tied
381 off throughout the process.

382 For sampling operations that require drilling, open boreholes and wells must be covered or
383 secured when unattended, including during crew breaks.

384 ELECTRICAL HAZARDS

385 Electric power often is supplied by gasoline or diesel engine generators. Working conditions may
386 be wet, and electrical shock with possibly fatal consequences may occur. In addition, it is
387 possible that drilling operations may encounter overhead or buried electrical utilities, potentially
388 resulting in exposure to very high voltages, which could be fatal or initiate fires.

389 All electrical systems used during field operations should be checked for proper grounding
390 during the initial installation. Temporary electrical power provided to the drill site shall be
391 protected by ground fault circuit interrupters.

392 NOISE HAZARDS

393 Power equipment is capable of producing sound levels in excess of 85dB(A), the eight-hour
394 threshold limit value recommended by the American Conference of Governmental Industrial
395 Hygienists (ACGIH). Exposure to noise levels in excess of 85dB(A) for long periods of time can
396 cause irreversible hearing loss. If noise levels
397 exceed 85dB(A), a controlled area must be
398 maintained at this distance with a posting at
399 each entrance to the controlled area to read:

CAUTION NOISE HAZARD Hearing Protection Required Beyond This Point
--

400 HEAT STRESS

401 The use of protective clothing during summer months significantly increases the potential for
402 personnel to experience heat stress. Adverse effects from heat stress include heat cramps,
403 dehydration, skin rash, heat edema, heat exhaustion, heat stroke or death. When heat stress
404 conditions exist, the following ought to be available:

- 405 • A cool and shaded rest area;
- 406 • Regular rest breaks;
- 407 • An adequate supply of drinking water; and
- 408 • Cotton coveralls rather than impermeable Tyvek coveralls.

409 CHEMICAL AND RADIOLOGICAL HAZARDS

410 The health and safety plan should contain information about a site's potential radionuclides and
411 hazards that might be encountered during implementation of field sampling and survey
412 procedures. All field personnel should read the health and safety plan and acknowledge an
413 understanding of the radiological hazards associated with a site. Site specific training must be
414 provided that addresses the chemical and radiological hazards likely to be associated with a site.
415 Field procedures should include either information relating to these hazards or should reference
416 appropriate sections of the Health and Safety Plan. References related to the use of protective
417 clothing are given in EPA (1987), DOE (1987, Appendix J), and in 29 CFR 1910, Subpart I.

418 When procuring environmental solid and liquid samples, unusual characteristics such as color,
419 suspended material, or number of phases and unusual odors should be noted and a description
420 should be provided to the on-site safety officer as well as the analytical laboratory. Additional
421 information concerning field methods for rapid screening of hazardous materials is presented in
422 EPA (1987). This source primarily addresses the appearance and presence of organic compounds
423 that might be present on occasions when one is collecting materials to detect radioactivity.
424 Checking samples for chemical or radiological hazards can be as simple as visual inspection or
425 using a hand-held radiation meter to detect radiation levels. Adjustments to laboratory
426 procedures, particularly those involving sample handling and preparation, can only be made
427 when pertinent field information is recorded and relayed to the project planner and to the
428 laboratory. In some cases, a laboratory might not have clearance to receive certain types of
429 samples (such as explosives or chemical agents) because of their content, and it will be necessary
430 to divert these samples to an alternate laboratory. It might be necessary to reduce the volume
431 sampled in order to meet shipping regulations if high concentrations of radioactivity are present
432 in the samples. In some cases, the activity of one radionuclide might be much higher than others
433 in the same sample. Adjustments made on the basis of the radionuclide of higher activity might
434 result in collection of too little of another radionuclide to provide adequate detection and thus
435 prevent identification of these radionuclides because of their relatively low minimum detectable

436 concentrations. These situations should be considered during planning and documented in the
437 appropriate sampling plan document.

438 10.2.11.2 Biohazards

439 Precautions should be taken when handling unknown samples in the field. Some examples are
440 wearing gloves, coveralls or disposable garments, plastic booties, dust masks or other respiratory
441 protection. Some biohazards may be snakes, ticks, spiders, and rodents (Hanta virus). Prevention
442 of potential exposure is the goal of a safety program. The type of protective equipment in the
443 field should be discussed in the planning process and specified in the appropriate plan document.
444 Since there are many specifics that are site dependent, it is difficult to create a comprehensive
445 list. But the information is discussed to provide an awareness and starting point for additional
446 discussion.

447 PERSONNEL TRAINING AND QUALIFICATION

448 All field operations that could lead to injury for sample collectors should be performed by
449 personnel trained to documented procedures. When sampling is conducted in radiologically
450 controlled areas (RCAs) as defined in regulatory standards (i.e., 10 CFR 20, 10 CFR 835).
451 Formal training and qualification of field personnel may be required.

452 Training may require both classroom and practical applications in order to familiarize personnel
453 with the basic theory of radiation and radioactivity and the basic rules for minimizing external
454 exposures through time, distance, shielding, and avoidance of internal exposure (by complying
455 with rules regarding smoking, drinking, eating, and washing of hands). Other topics to cover
456 include common routes of exposure (e.g., inhalation, ingestion, skin contact); proper use of
457 equipment and the safe handling of samples; proper use of safety equipment such as protective
458 clothing, respirators, portable shielding, etc.

459 Guidance for the training and qualification of workers handling radioactive material has been
460 issued by the Nuclear Regulatory Commission (see appropriate NRC NUREGs and Regulatory
461 Guides on training of radiation workers), Department of Energy (1994), and the Institute of
462 Nuclear Power Operations (INPO 88-010). These and other documents should be consulted for
463 the purpose of training and qualifying field personnel.

464 PERSONNEL MONITORING AND BIOASSAY SAMPLING

465 When conditions dictate the need for personnel monitoring, various methods are commonly
466 employed to assess external and internal exposure that might have resulted from the inhalation or
467 ingestion of a radionuclide.

468 To monitor for external exposures to the whole body or extremities, thermoluminescent
469 dosimeters (TLDs) or film badges may be used to document a worker's exposure. For internal
470 exposures, assessment of dose may be based on: (1) air monitoring of the work area or the
471 worker's breathing zone; (2) *in vivo* bioassay (whole-body counting); or (3) *in vitro* bioassays
472 that normally involve urinalysis but may also include fecal analysis and nasal smears. For *in vitro*
473 bioassays (i.e., urine or fecal), the standard method involves a 24-hour sample collection in a
474 sealable container. Samples may be kept under refrigeration until laboratory analysis can be
475 performed to retard bacterial action. (Bioassay sample collection is normally not performed in the
476 "field.")

477 The following guidance documents may be used for personnel monitoring and the collection and
478 preservation of bioassay samples:

- 479 • ANSI/ANS HPS N13.30 (1996), Performance Criteria for Radiobioassay;
- 480 • ANSI/ANS HPS N13.14 (1994), Internal Dosimetry Programs for Tritium Exposure—
481 Minimum Requirements;
- 482 • ANSI/ANS HPS 13.22 (1995), Bioassay Programs for Uranium;
- 483 • ANSI/ANS HPS 13.42 (1997), Internal Dosimetry for Mixed Fission Activation Products;
- 484 • DOE Implementation Guide, Internal Dosimetry Program, G-10 CFR 835/C1—Rev. 1 Dec.
485 1994a;
- 486 • DOE Implementation Guide, External Dosimetry Program, G-10 CFR 835/C2—Rev. 1 Dec.
487 1994b;
- 488 • DOE Implementation Guide, Workplace Air Monitoring, G-10 CFR 835/E2—Rev. 1 Dec.
489 1994c;
- 490 • DOE Radiological Control Manual, DOE/EH-0256T, Rev. 1, 1994d;
- 491 • NRC Regulatory Guide 8.9, Acceptable Concepts, Models, Equations, and Assumptions for a
492 Bioassay Program;
- 493 • NRC Regulatory Guide 8.11, Applications of Bioassay for Uranium;
- 494 • NRC Regulatory Guide 8.20, Applications of Bioassay for ¹²⁵I and ¹³¹I;
- 495 • NRC Regulatory Guide 8.22, Bioassays at Uranium Mills;
- 496 • NRC Regulatory Guide 8.26, Applications of Bioassay for Fission and Activation Products;
- 497 • NRC Regulatory Guide 8.32, Criteria for Establishing a Tritium Bioassay Program;
- 498 • NCRP (1987), Use of Bioassay Procedures for Assessment of Internal Radionuclides
499 Deposition; and
- 500 • INPO (1988), Guidelines for Radiological Protection at Nuclear Power Stations.

501 **Part II: Matrix-Specific Issues That Impact Field Sample Collection,** 502 **Processing, and Preservation**

503 Field processing should be planned in advance so that all necessary materials are available during
504 field work. Preparing checklists of processing equipment, instruments, and expendable
505 materials—as exemplified in part by lists accompanying sampling procedures described by EPA

506 1994—helps this planning effort and serves to organize field methods. Field personnel who
507 communicate problems should prevent loss of time, effort, and improper sample collection, as
508 well as documents exactly what equipment, instruments, etc. were used.

509 The initial steps taken in the field frequently are critical to the laboratory analysis performed
510 hours, days, or even weeks after a sample is obtained. Various sample preparation steps may be
511 required before samples are packaged and shipped for laboratory analysis. The need for sample
512 processing and preservation is commonly determined by the sample matrix, the data quality
513 objectives of the analysis, the nature of the radionuclide, and the analytical method.

514 The goal of sample preservation is to maintain the integrity of the sample between the time the
515 sample is collected and the time it is analyzed, thus assuring that the analysis is performed on a
516 sample representative of the media collected. In general, the aim of sample preservation is to
517 limit biological and chemical actions that might alter the concentration or physical state of the
518 radionuclide constituents or analytes. For example, cations at very low concentrations can be lost
519 from solution (e.g., cesium can exchange with potassium in the glass container, and radio-
520 nuclides can be absorbed by algae or slime growths in sample lines or containers that remain in
521 the field for extended periods). Requirements for sample preservation should be determined
522 during project planning when analytical protocols are selected. Sample preservation in the field
523 typically follows or accompanies processing activities.

524 This section provides matrix-specific guidance that focuses on the preparation and processing of
525 field samples. In order to assist project planners in developing a sampling plan, a limited
526 discussion is also provided that describes matrix-specific methods commonly employed for the
527 collection of field samples. Guidance is presented for only the most common materials or
528 environmental media, which are generically classified as liquids, solids, and air. In some
529 instances, a solid material to be analyzed involves particulate matter suspended in a liquid or air
530 that is commonly obtained by filtration. Because filter media can affect analytical protocols, a
531 separate discussion is provided that addresses sample materials contained on filter materials,
532 including surface contamination associated with wipe samples.

533 **10.3 Liquid Samples**

534 Liquid samples are typically classified as aqueous, non-aqueous, and as mixtures. Aqueous
535 samples requiring analysis are likely to represent surface water, ground water, drinking water,
536 precipitation, tanks and lagoons, and runoff. Non-aqueous liquids may include a variety of
537 solvents, oils and other organic liquids. Mixtures of liquids represent a combination of aqueous
538 and non-aqueous liquids or a solid suspended in either aqueous and non-aqueous liquids.
539 Standardized water sampling procedures are described in numerous documents (APHA, 1996;
540 EPA, 1985; EPA, 1987; DOE, 1997; ASTM D3370). Important decisions include the choice of
541 instrument or tool used to obtain the sample, the sample container material, the need for sample
542 filtration, and the use of sample preservatives.

543 **10.3.1 Liquid Sampling Methods**

544 The effect of the sample collection process on the sample integrity needs to be understood and
545 managed. Two examples are dissolved gases and cross contamination. It may be necessary to
546 minimize dissolved oxygen and carbon dioxide which may cause some dissolved metals to
547 undergo reaction or precipitation.

548 Sampling is discussed in Navy Environmental Compliance Sampling and Field Testing
549 Procedures Manual, NAVSEA T0300-AZ-PRO-010. USACE discusses sampling in *Technical*
550 *Project Planning Guidance for Hazardous, Toxic and Radioactive Waste (HTRW) Data Quality*
551 *Design, Engineer Manual EM-200-1-2, Appendix H, Sampling Methods, July 1995. This*
552 *reference has been superseded but the revision does not include sampling. The sampling*
553 *references listed in Appendix H are:*

- 554 • U.S. Environmental Protection Agency (EPA). 1984. Characterization of Hazardous Waste
555 Sites—A Method Manual, Vol. II, Available Sampling Methods, Second Edition, EPA 600-
556 4-84-076.
- 557 • U.S. Environmental Protection Agency (EPA). 1982. Handbook for Sampling and Sample
558 Preservation of Water and Wastewater, EPA 600-4-82-029.
- 559 • U.S. Environmental Protection Agency (EPA). 1986. Compendium of Methods for
560 Determination of Superfund Field Operation Methods, EPA 600-4-87/006.
- 561 • U.S. Environmental Protection Agency (EPA). 1987. A Compendium of Methods for
562 Determination of Superfund Field Operation Methods, EPA 540-P-87-001a, OSWER
563 Directive 9355.0-14.
- 564 • U.S. Department of the Interior. 1980. National Handbook of Recommended Methods for
565 Water for Water-Data Acquisition, Volume I and II.

566 **10.3.2 Liquid Sample Preparation: Filtration**

567 Filtration of a water sample may be a key analytical planning issue and is discussed in Chapter 3,
568 Section 3.3.2. A decision needs to be made during project planning whether or not to filter the
569 sample in the field. Filtration of water or other liquids may be required to determine contaminant
570 concentrations in solubilized form, suspended particulates, or sediment. The method of filtration
571 will depend on the required sample volume, the amount and size of suspended particulates, and
572 the availability of portable equipment and resources (e.g., electricity).

573
574 The potential need to filter a water sample principally depends on the source of water and the
575 objectives of the project investigation. If, for example, the source of water is drinking water “at-

576 the-spigot” and the intent is to assess human internal exposure from ingestion, unfiltered tap
577 water samples are likely to be required. Conversely, filtration may be required for water taken
578 from an unlined field monitor well that is likely to contain significant amounts of particulate
579 matter. These solids are of little relevance but may interfere with radioanalytical protocols (e.g.,
580 sample absorption may occur during gross alpha or beta counting where the analytical procedure
581 involve s the simple evaporation of a water aliquant on a planchet).

582 For remote sampling sites, sample processing may be restricted to gravity filtration that requires a
583 minimum of equipment and resources. Drawing samples through filters by pressure or suction
584 that is created by syringe, vacuum pump, or aspiration are alternative options. If filter papers or
585 membranes capture materials that will be retained for analysis, they should be handled with clean
586 rubber or plastic gloves, forceps, or other instruments to prevent sample contamination.

587 Each Federal Agency may have unique guidance to determine the need and process for filtering
588 samples. One performance-based example is that of EPA, discussed in the next section. This
589 guidance applies to either the field or laboratory filtration.

590 10.3.2.1 EPA Guidance for Samples/Filtration

591 The Special Topics Subcommittee of EPA’s Science Advisory Board’s Environmental
592 Engineering Committee met to examine the question of whether or not to filter ground-water
593 samples when analyzing for metals in the context of a review of the Office of Emergency and
594 Remedial Response’s (OERR) proposed guidance on field filtration of ground-water samples
595 taken from monitoring wells for metals analysis as part of a Superfund site assessment (EPA,
596 1997). The key findings of the Subcommittee were:

- 597 • Several factors could introduce errors in the sampling and analysis of ground water for metals
598 or metallic radionuclides. Well construction, development, sampling, and field filtering are
599 among the steps that could influence the metals measured in the ground-water samples. Field
600 filtering is often a smaller source of variability and bias compared to these other factors.
601 Therefore, the Agency should emphasize in its guidance the importance of proper well
602 construction, development, purging, and water pumping rates so that the field filtering
603 decisions can also be made accurately.

- 604 • Under ideal conditions, field-filtered ground-water samples should yield identical metals
605 concentrations when compared to unfiltered samples. However, under non-ideal conditions,
606 the sampling process may introduce geological materials into the sample and would require
607 field filtration. Under such conditions, filtering to remove the geological artifacts has the
608 potential of removing colloids (small particles that may have migrated as suspended materials
609 that are mobile in the aquifer). Available scientific evidence indicates that when wells have
610 been properly constructed, developed, and purged, and when the sample has been collected
611 without stirring or agitating the aquifer materials (turbidity less than 5 nephelometric turbidity

612 units, NTU), then field filtering should not be necessary. For Superfund site assessments, the
613 low-flow sampling technique without filtration is the preferred sampling approach for
614 subsequent metal analysis when well construction, well maintenance, and hydrogeological
615 conditions such as flow rate allow. Under such conditions, the collected samples should be
616 representative of the dissolved and particulate metals that are mobile in ground-water
617 systems. The Agency's proposal to rely on low flow sampling and unfiltered samples is a
618 conservative approach that favors false positives over false negatives.

- 619 • When the turbidity of the sample is high, the situation is different. In-line filtering provides
620 samples that retain their chemical integrity. Therefore, field filtering of properly collected
621 ground-water samples should be done when turbidity in the samples is higher than 5 NTU,
622 even after slow pumping has been utilized to obtain the sample.

623 They acknowledged, however, that differences in the way wells are installed, their packing
624 materials, and the techniques used to collect ground-water samples can lead to variability in
625 analytical results between wells and between individual samples. Filtering a sample can be seen
626 as a way to remove suspended particles and some colloids that contain metals that would not
627 normally be in the ground water if the material were not disturbed during sampling. Here a
628 colloid is defined as a particle that ranges in size from 0.003 to 10 μm (Puls et al., 1990) or
629 particles having diameters of less than 10 μm (Puls and Powell, 1992). The literature indicates
630 that colloids as large as 2 μm can be mobile in porous media (Puls and Powell, 1992), and that
631 colloid concentration can be as high as 1,000 times higher in fractured granitic systems
632 (McCarthy and Deguelde, 1993). Saar (1997) presents a review of the industry practice of
633 filtration of ground-water samples. For some sites with low hydraulic conductivity the presence
634 of an excess of colloids presents numerous monitoring challenges and field filtration might be
635 necessary.

636 The desire to disturb the aquifer as little as possible has led to the use of low-flow sampling of
637 wells—low-flow purging and sampling occurs typically at 0.1 to 0.3 L/min (Saar, 1997). The
638 low-flow technique maximizes representativeness by (EPA, 1997):

- 639 • Minimizing disturbances that might suspend geochemical materials that are not usually
640 mobile;
- 641 • Minimizing disturbances that might expose new reactive sites that could result in leaching or
642 adsorption of inorganic constituents of ground water;
- 643 • Minimizing exposure of the ground water to the atmosphere or negative pressures, ensuring
644 that the rate of purging and sampling does not remove ground water from the well at a rate
645 much greater than the natural ground-water influx; and

- 646 • Monitoring indicator parameters to identify when stagnant waters have been purged and the
647 optimum time for sample collection.

648 In summary, based on the ability of the low-flow sampling technique to collect representative
649 samples, EPA suggests that filtering of ground-water samples prior to metals analysis is usually
650 not required (EPA, 1997).

651 10.3.2.2 Filters

652 When filtration is required, it should be done in the field or as soon as practicable. The
653 advantages of filtering in the field are that acid preservatives can be added shortly thereafter
654 which minimizes both the adsorption of soluble contaminants and avoids the dissolution of
655 particulate matter, volume reduction, and waste reduction. Unless specific requirements dictate
656 otherwise, the removal of suspended particles is commonly achieved by filtration that removes
657 particles larger than 0.45 μm (ASTM D3977).

658 In other instances, the investigative objectives may not be restricted to water-solubilized
659 contaminants but include analysis of contaminated suspended particulate matter. To detect the
660 presence of radionuclides that are highly insoluble, such as isotopes of uranium, thorium, and
661 plutonium, analysis of particulate matter is considered more sensitive than the filtered water
662 (EPA, 1994).

663 The fact that small particles pass through membrane filters has been recognized for some time
664 (Kennedy et al., 1974). The arbitrary cutoff of 0.45 μm between dissolved and suspended matter
665 has gained such wide use that it is the filter size that is commonly recommended by laboratory
666 protocols. Filtering through a 0.45 μm filter may take considerable time and may require suction
667 or pressure to accomplish in a reasonable time.

668 It should be noted, however, that manufacturers of filters usually specify only what will not pass
669 through the filter; they make no claims concerning what actually does pass through the filter.
670 Laxen and Chandler (1982) present a comprehensive discussion of some effects of different filter
671 types. They refer to thin (5 to 10 μm) polycarbonate filters as screen types, and thick (100 to
672 150 μm) cellulose nitrate and acetate filters as depth types. The polycarbonate-screen type clogs
673 much more rapidly. Once the filtration rate drops, particles that would normally pass through the
674 filter are trapped in the material already retained. Hence, the use of so-called polycarbonate-
675 screen filters, because of their increased propensity to clog, is generally not recommended.
676

677 In addition to the difficulty of contending with clogging, Silva and Yee (1982) report adsorption
678 of dissolved radionuclides on membrane filters. Although these drawbacks cannot be completely
679 overcome, they are still less than the potential difficulties that arise from not filtering.

680 Finally, good laboratory practices must be used for field sampling. The most likely sources of
681 contamination for the filters are improperly cleaned tubing and filter holders and handling the
682 filters with contaminated fingers. Tubing and holders should be thoroughly cleaned and rinsed
683 between samples and the entire system should be rinsed several times with the water to be
684 sampled. Filters should be handled with clean rubber gloves.

685 **10.3.3 Field Preservation of Liquid Samples**

686 Sample degradation may occur between the time of collection and analysis due to microbial
687 contaminants or chemical interactions. Although sample degradation cannot destroy or alter the
688 radiological properties of a contaminant, it can alter the radionuclide's chemical properties and
689 its potential distribution within a sample. For example, microbial processes are known to affect
690 both the chemical state and the distribution of radioelements due to oxidation-reduction
691 reactions, complexation and solubilization by metabolic compounds, bioaccumulation,
692 biomylation, and production of gaseous substances such as CO₂, H₂, CH₄, and H₂S (Francis,
693 1985; Pignolet et al., 1989).

694 10.3.3.1 Sample Acidification

695 Acidification is the method of choice for preserving most types of water samples. The principal
696 benefit of acidification is that it keeps many radionuclides in solution and minimizes their
697 potential for removal by chemical and physical adsorption or by ion exchange. The mode by
698 which a radionuclide is potentially removed from solution is strongly affected by the radionuclide
699 and the container material. For example, studies conducted by Bernabee et al. (1980) and Milkey
700 (1954) demonstrated that the removal of metal ions from solution is dominated by physical (i.e.,
701 van der waals) adsorption. Their conclusion is based on: (1) their observation that the loss of
702 uranium, lead, and thorium ions from solution was significantly greater for containers made of
703 polyethylene when compared to borosilicate glass; and (2) the fact that while adsorption by glass
704 may potentially involve all three adsorption processes; with polyethylene plastic, there are no
705 valence-type attractive forces or ions to exchange and only physical van der waals adsorption is
706 possible.

707 Similar observations were reported by: (1) Dyck (1968), who compared long-term adsorption of
708 silver ions by molded plastic to glass containers; (2) Jackson (1962), who showed that
709 polyethylene containers absorbed about five times as much ⁹⁰Sr as glass containers at pH of about
710 seven; and (3) Martin and Hylko (1987a; 1987b), who reported that greater than 50 percent of
711 ⁹⁹Tc was adsorbed by polyethylene containers from non-acidified samples.

712 For sample acidification, either nitric or hydrochloric acid is commonly added until a pH of less
713 than two. Table 7010:1 in *Standard Methods for the Examination of Water and Wastewater*
714 (APHA, 1995) and Method 900.0 in *Prescribed Procedures for Measurement of Radioactivity in*

715 *Drinking Water* (EPA, 1980) provide additional guidance. Guidance for sample preservation by
716 acidification has been issued by Federal Agencies and others as summarized below.

717 In instances of very low activity samples where container adsorption poses a significant concern,
718 but where acidification of the sample interferes with the radioanalytical method, the choice of
719 sample container may be limited to glass or require alternative methods. For example, the use of
720 acids as a preservative is not recommended for the analysis of tritium (^3H), carbon-14 (^{14}C), or
721 radon in water, and precautions must be taken for the following reasons:

- 722 • For radon, sample preservation offers no benefit and is therefore not required for analytical
723 accuracy.
- 724 • The addition of acid to a sample containing ^{14}C may result in the production of $^{14}\text{CO}_2$ and the
725 loss of radioactivity from the sample.
- 726 • The adverse impact of acid on tritiated water is due to the fact that water dissociates and
727 recombines continuously (i.e., $\text{H}_2\text{O} \rightleftharpoons \text{H}^+ + \text{OH}^-$ or $\text{HO} \rightleftharpoons \text{T}^+ + \text{OH}^-$). The tritium ion that was
728 part of the water molecule may, therefore, be exchanged for the hydrogen ion from the acid.
729 The impact of this exchange is realized as a result of distillation, which is a common method
730 for purifying water in preparation for liquid scintillation counting. When the sample is heated
731 and the distillate is collected on a cold finger, distilled tritiated water, in the presence of acid,
732 would have a reduced specific activity over the original sample.

733 Although acidification has been shown to effectively reduce the adsorption of technetium by
734 polyethylene, technetium in the TcO_4^- state has been observed to volatilize in strong acid
735 solutions during evaporation while preparing water samples for gross beta analysis (NAS, 1960).
736 To hasten evaporation, the planchet is commonly flamed. This dilemma can be resolved by either
737 precoating planchets with a film of detergent prior to the addition of the acidified water sample
738 or by passive evaporation of the acidified water sample that avoids the higher temperature
739 associated with flaming (Blanchard et al., 1993).

740 10.3.3.2 Non-Acid Preservation Techniques

741 If a sample contains significant organics, or if contaminants under investigation react with acids
742 that interfere with the radioanalytical methods, other methods of sample preparation should be
743 considered.

744 REFRIGERATION AND FREEZING

745 The effect of refrigeration or freezing temperatures to arrest microbial activity is a fundamental
746 concept. Temperatures near the freezing mark or below not only retard or block bacterial growth
747 but arrest essentially all other metabolic activity. It should, however, be noted that most bacteria

748 can survive even in extreme temperatures. (Indeed, if a suspension of bacterial cells is frozen
749 rapidly with no appreciable formation of ice crystals, it can be kept at temperatures as low as
750 -194° C for indefinite periods of time with little loss of viability.)

751 The choice between refrigeration and freezing is dictated by the potential impacts of ice
752 formation on sample constituents. Besides physical changes of organic constituents, the initial
753 formation of ice crystals and the exclusion of any solutes may concentrate the solutes to the point
754 of precipitation. Quick freezing methods that minimize ice crystal formation are beneficial for
755 preserving some organic constituents. Quick freezing is commonly done by packing sealed
756 samples in liquid nitrogen or dry ice. Care must be taken, however, to avoid container breakage
757 due to sample volume expansion. An air space of a least 10 percent and a container made of
758 plastic provide reasonable assurance for container integrity.

759 When refrigeration is employed, attempts should be made to avoid temperatures that could result
760 in slow freezing and the formation of ice crystals. Optimum refrigeration temperatures for sample
761 preservation at $4 \pm 2^\circ$ C can be achieved by packing samples in ice or freeze packs within a
762 thermally insulated leak-proof container (ASTM D3856; ASTM D3370).

763 PAPER PULP

764 Adsorption and loss of radionuclides over time to the container wall can be avoided with the
765 addition of paper pulp. Due to its adsorptive property and large surface, paper pulp has been
766 shown to remove more than 95 percent of radionuclides from solution (Bernabee et al., 1980).
767 About two grams of finely ground paper pulp are added per liter of acidified sample at time of
768 collection. The pH should be adjusted to one or less and vigorously shaken. The sample may be
769 stored in this condition for an extended period of time. To prepare for analysis, the pulp is
770 removed from solution by filtration and subjected to wet ashing using strong acids (Chapter 12).
771 This ashed solution is commonly added to the original filtrate to make a reconstituted sample
772 solution.

773
774 The use of paper pulp and the need for wet ashing, however, pose problems for certain
775 radioanalytical laboratory protocols and must therefore be thoroughly evaluated.

776 SULFITE

777 To prevent the loss of radioiodine from solution, sodium bisulfite (NaHSO_3) or sodium
778 metabisulfite ($\text{Na}_2\text{S}_2\text{O}_5$) may be used. These compounds act as strong reducing agents and
779 prevent the volatilization of iodine. If acid is also employed to preserve samples for analysis of
780 other radionuclides, it is important to note that acid will negate the reductant's effectiveness in
781 behalf of iodine. For this reason, samples collected for iodine analyses typically are collected and
782 preserved in a separate container. It should also be noted that the reducing environment produced
783 by the sulfite preservative may render iron, uranium, and other easily reduced elements or their

784 compounds to an insoluble state. The loss of reduced insoluble radionuclides from solution will
 785 have an obvious adverse impact on radioanalytic measurements that require chemical separation.
 786 Chapter 14.9 has additional information on carriers and tracers.

787 **OTHERS**

788 Other methods that have been used to preserve liquid samples containing organics and biological
 789 materials include chemical preservatives (e.g., formaldehyde and methanol) or quick freezing by
 790 means of liquid nitrogen. Table 10.1 summarizes the advantages and disadvantages of these and
 791 previously described preservation methods.

TABLE 10.1—Summary of sample preservation techniques.

Preservation Technique	Advantages	Disadvantages
Addition of HNO ₃	Reduces pH and inhibits plating of metals on container walls.	Strong oxidizer that might react with organic compounds. Tritium might be separated preferentially as acid hydrogen; ¹⁴ C might be lost as ¹⁴ CO ₂ .
Addition of Hcl	Reduces pH and inhibits plating of metals on container walls. Chloride forms strong anionic complexes with Iron and Uranium.	Tritium will be preferentially separated as acid hydrogen; ¹⁴ C might be lost as ¹⁴ CO ₂ . Might cause corrosion of stainless steel planchets on gross analyses.
Addition of Sulfite	Forms a reducing environment to prevent the volatilization of iodine.	Might produce insoluble compounds from reduced forms of iron or uranium.
Addition of Formaldehyde	Preserves organic samples. Prevents further biological activity.	May create disposal problems.
Cooling (Ice at approximately 0° C)	Preserves organic samples (i.e., water, foods). Reduces dehydration and retains moisture. Reduces biological activity.	Ice melts, requiring replacement over time.
Freezing (Dry Ice at approximately -78° C)	Preserves organic samples (i.e., water, plant, animal). Suspends biological activity.	Dry ice sublimates and requires replacement.
Addition of Paper Pulp	Provides large surface area for adsorption of metals, thus minimizing adsorption on container walls.	Requires pH to be one or less. Requires filtration and wet ashing of paper pulp and combining liquids to make a new solution.

806 **10.3.4 Liquid Samples: Special Cases**

807

808 In some cases, liquid samples require special handling in order to preserve or retain a volatile or
809 gaseous radionuclide. The following are examples of specific methods used to recover or
810 preserve such samples of interest.

811 10.3.4.1 Radon-222 in Water

812 Waterborne radon is analyzed most commonly by liquid scintillation methods, although gamma
813 spectroscopy and other methods have been employed or proposed. Liquid scintillation has the
814 obvious advantage of being designed for automated sample processing and is, therefore, less
815 labor intensive or costly. A key to consistency in analytical results is the zero headspace sampling
816 protocol such as the one described below.

817 Since radon is inert and nonpolar, it diffuses through plastic more rapidly than glass. The use of
818 plastic scintillation vials, therefore, leads to significant loss of radon in water (Whittaker, 1989;
819 Hess and Beasley, 1990). For this reason, it is recommended that the water sample is collected in
820 a 23 mL glass scintillation vial, capped with a Teflon or foil-lined cap.

821 Samples are collected from a non-aerated faucet or spigot, which has been allowed to flow for
822 sufficient time so that the sample is representative of the water in the distribution system or well.
823 The time will vary depending on the source. The following zero headspace procedure will
824 minimize the loss of radon from the sample during collection:

- 825 • Place sample vial in a 300-600 mL beaker or other suitable container and attach the universal
826 adapter and fill-line to spigot, and start the flow.
- 827 • Fill the vial to prevent it from floating. Then fill the beaker until the vial is submerged.
- 828 • Place the tip of the fill line about two thirds of the way into the vial and fill until
829 approximately two or more vial (50-100 mL) volumes have been displaced.
- 830 • Carefully remove the vial with a pair of 10-inch tweezers and cap the vial with a Teflon or
831 foil-lined cap. Invert the sample and check for air bubbles. If any bubbles are present, discard
832 the sample and repeat the sampling procedure. Record date and time the sample was collected
833 and store the sample in a cooler to prevent temperature excursions. Transport the samples to
834 the laboratory in a cooler or other suitable insulated package.

835 10.3.4.1 Milk

836 Milk commonly is viewed as the food product of greatest potential dose significance for airborne
837 releases of radionuclides. Due to the metabolic discrimination, however, only a few radionuclides

838 have a significant dose impact via the milk pathway, notably ⁹⁰Sr, ¹³¹I, and ¹³⁷Cs. Raw milk
839 should be obtained from the closest cows or goats downwind from a source.

840 To prevent milk from souring or curdling, samples should be refrigerated. Preservation of milk
841 may also be achieved through the addition of formaldehyde or methanol (DOE, 1987),
842 merthiolate, or Thimerosal (EPA, 1994). Analytical procedures for select radionuclides in milk
843 are well established and should be considered when deciding on a sample preservation method.

844 Owing to the volatility and potential loss of ¹³¹I, a known amount of NaI dissolved in water
845 should be added to the milk sample at time of collection. The NaI not only serves as a carrier for
846 the chemical separation of radioiodine, but also provides a quantitative tool for determining any
847 loss prior to analysis (DOE, 1990).

848 **10.3.5 Non-aqueous Liquids and Mixtures**

849 Non-aqueous liquids and mixtures include a wide range of organic fluids or solvents, organic
850 materials dissolved in water, oils, lubricants, etc. These liquids are not likely to represent
851 contaminated environmental media or matrices, but most likely represent waste streams that must
852 be sampled. Non-aqueous waste streams are generated as part of normal operations by nuclear
853 utilities, medical facilities, academic and research facilities, State and Federal Agencies, radio-
854 pharmaceutical manufacturers, DOE weapons complexes, mining and fuel fabrication facilities,
855 etc. Examples of these non-aqueous liquids and mixtures include waste oils and other lubricants
856 that are generated routinely from maintenance of various types equipment associated with
857 nuclear power plant operations or the production of nuclear fuel and nuclear weapon
858 components; and organic and inorganic solvents, acids, and bases that are used in a variety of
859 medical, research, and industrial applications.

860 In addition to the production of non-aqueous liquid wastes from routine operations by these
861 facilities, large quantities of non-aqueous liquids containing radionuclide contaminants are also
862 generated by routine facility decontamination efforts and final decontamination associated with
863 facility decommissioning. For decontamination and decommissioning activities, a wide range of
864 processes have been developed that employ halogenated organic compounds, such as Freon,
865 chloroform, or trichloroethane. Other aggressive chemical decontamination processes involve
866 dissolution and removal of metal and oxide layers from surfaces using acid solutions (e.g.,
867 sulfuric acid, nitric acid, phosphoric acids, and oxalic acid). Chemical decontamination also may
868 use chelating agents in concentrated processes (5 to 25 percent wt. chemical in solution) and
869 dilute processes (one percent wt. or less chemicals in solution). Examples of chemical processes
870 that can be used in both concentrated and dilute forms include the low oxidation-state transition-
871 metal ion (LOMI) and LOMI-nitric permanganate, developed by Dow Chemical Company and
872 AP/Citron. The reagents used in both the concentrated and dilute processes include chelating and
873 complexing agents such as ethylene diamine tetra acetic acid (EDTA), diethylene triamine penta-
874 acetic acid (DTPA), citric acid, oxalic acid, picolinic acid, and formic acid. Chelating agents and

875 organic acids are used in decontamination formulas because they form strong complexes with
876 actinides, lanthanides, heavy metals, and transition metals and assist in keeping these elements in
877 solution.

878 Generally, these chemical decontamination solutions, once used, are treated with ion-exchange
879 resins to extract the soluble activity. The ion-exchange decontamination solutions must,
880 nevertheless, be sampled to assess the amount of residual radioactivity.

881 The radionuclides that may be encountered with non-aqueous liquids and mixtures depend on
882 both the nature of the liquid and its usage. The following listing of radionuclides and liquids are
883 based on published data collected by NRC (1992) and the State of Illinois (Klebe 1998; IDNS
884 1993-1997):

885 • Toluene/xylene/scintillation fluids used by research and clinical institutions: ^3H , ^{14}C , ^{32}P , ^{35}S ,
886 ^{45}Ca , ^{63}Ni , ^{99}Tc , ^{90}Sr , ^{125}I , ^{147}Pm , $^{226/228}\text{Ra}$, $^{228/230/232}\text{Th}$, $^{234/235/238}\text{U}$, $^{238/239/241}\text{Pu}$, ^{241}Am .

887 • Waste oils and lubricants from operation of motors, pumps, and other equipment: ^3H , ^{54}Mn ,
888 ^{65}Zn , ^{60}Co , $^{134/137}\text{Cs}$, $^{228/230/232}\text{Th}$.

889 • Halogenated organic and solvents from refrigeration, degreasing, and decontamination: ^3H ,
890 ^{14}C , ^{32}P , ^{35}S , ^{54}Mn , $^{58/60}\text{Co}$, ^{63}Ni , ^{90}Sr , $^{125/129}\text{I}$, $^{134/137}\text{Cs}$, $^{226/228}\text{Ra}$, $^{228/230/232}\text{Th}$, $^{232/234/238}\text{U}$,
891 $^{238/239/241}\text{Pu}$, U-nat.

892 • Other organic solvents from laboratory and industrial operations and cleaning: ^3H , ^{32}P , ^{35}S ,
893 ^{45}Ca , ^{125}I , U-nat.

894 • Inorganic and organic acids and bases from extraction processes and decontamination: ^3H ,
895 ^{14}C , ^{32}P , ^{35}S , ^{54}Mn , ^{67}Ga , ^{125}I , ^{60}Co , ^{137}Cs , $^{201/202}\text{Th}$, and U-nat.

896 Due to the large number of potential non-aqueous liquids and the complex mixtures of
897 radionuclide contaminants that may require radiochemical analysis, a comprehensive discussion
898 of sample preparation and preservation is beyond the scope of this discussion. In most instances,
899 however, these samples are not likely to require refrigeration or chemical preservatives that
900 protect against sample degradation.

901 Some organic solvents and highly acidic or basic liquids may react with plastic containers,
902 causing brittleness or breakage. In selecting sample containers for these non-aqueous samples, it
903 is important to assess the manufacturer's product specifications, which typically provide
904 information regarding the container's resistance to chemical and physical agents. When non-
905 aqueous samples are stored for long periods of time, containers should be checked routinely.

906 **10.4 Solids**

907
908 Solid samples consist of a wide variety of materials that include soil and sediment, plant and
909 animal tissue, metal, concrete, asphalt, trash, etc. In general, most solid samples do not require
910 preservation, but require specific processing in the field before transporting to the laboratory for
911 analysis. For example, soil sample field processing may require sieving in order to establish
912 sample homogeneity. These and other specific handling requirements are described below in the
913 section on each type of solid sample.

914 The most critical aspect is the collection of a sufficient amount of a representative sample. One
915 purpose of soil processing is to bring back only that sample needed for the laboratory. Unless
916 instructed otherwise, samples received by the laboratory are typically analyzed exactly as they are
917 received. This means that extraneous material should be removed at the time of sample
918 collection, if indicated in the appropriated plan document.

919 In many instances, sample moisture content at the time of collection is an important factor. Thus,
920 the weights of solid samples should be recorded at the time a sample is collected. This allows one
921 to track changes in wet weight from field to laboratory. Dry and ash weights generally are
922 determined at the laboratory.

923 Unlike liquid samples that may be introduced or removed from a container by simple pouring,
924 solid samples may require a container that is designed for easy sample placement and removal.
925 For this reason, large-mouth plastic containers with screw caps or individual boxes with sealable
926 plastic liners are commonly used. The containers also minimize the risk for breakage and sample
927 cross-contamination.

928 **10.4.1 Soils**

929
930 ASTM D653 (*Standard Terminology Relating to Soil, Rock, and Contained Fluids*) defines soil
931 as: “Sediments or other unconsolidated accumulations of solid particles produced by the physical
932 and chemical degradation of rocks, and that might or might not contain organic matter.” ASTM
933 C999 provides generic guidance for soil sample preparation for the determination of
934 radionuclides. The American Society for Testing and Materials provides additional information
935 on soil and rock in the following standards:

- 936
- ASTM D 4914, Section 4, Construction, Volume 4.08 Soil and Rock (I).
 - ASTM D 4943, Section 4, Construction, Volume 4.09 Soil and Rock (II): Geosynthetics.
- 937

938 The distribution of radionuclides in soil should be assumed to be heterogeneous. The degree of
939 heterogeneity is dictated by the radionuclide’s mode of entry into the environment and soil, the
940 chemical characteristics of the radionuclide contaminant, soil composition, meteorological and
941 environmental conditions, and land use. For example, soil contamination from an airborne
942 release of a radionuclide with strong affinity for clay or other mineral constituents of soil (i.e.,

943 high k_d value) will likely exhibit a gradient with rapidly diminishing concentrations as a function
944 of soil depth. Moreover, contamination may be differentially distributed among soil particles of
945 different sizes. In most cases, because the contaminant is adsorbed at the surface of soil particles
946 and since the surface-to-volume ratio favors smaller particles, smaller soil particles will exhibit a
947 higher specific activity when compared to larger particles. If land areas include areas of farming,
948 tilling of soil will clearly impact the distribution of surface contamination.

949 10.4.1.1 Soil Sample Preparation

950 Extraneous material should be removed at the time of sample collection, if indicated in the
951 appropriate plan document. The material may have to be saved and analyzed separately,
952 depending on the project requirements and MQOs. If rocks, debris, and roots are removed from a
953 soil sample after it arrives at the laboratory, there might not be sufficient material to complete all
954 the requested analyses. A sufficient amount of sample should be collected to provide the net
955 quantity necessary for the analysis. Subsequent drying at the laboratory may remove a large
956 percentage of the sample weight that is available for analysis. Field-portable balances or scales
957 may be used to weigh samples as they are collected, further ensuring sufficient sample weights
958 are obtained. For certain types of samples, the project DQOs may require maintaining the
959 configuration of the sample, such as core samples where concentration verses depth will be
960 analyzed.

961 The project plan should address the impact of heterogeneity of radionuclide distribution in soil.
962 Some factors to consider that may impact radionuclide distribution are: determining sampling
963 depth, the need for removal of vegetative matter, rocks, and debris, and the homogenation of soil
964 particulates. For example, soil sampling depths of the top 5 cm is recommended for soils
965 contaminated by recent airborne releases (ASTM C998); soil depth to 15 cm may be appropriate
966 when exposure involves the need to monitor the root zone of food crops (MARSSIM, 2000;
967 NRC, 1990). The need for sample field QC, such as field splitting, should be evaluated. Some
968 types of field QC can be used to evaluate the extent of radionuclide homogeneity. In general, no
969 special preservation measures are required for soil samples; however, preliminary soil sample
970 preparation involving drying, sieving, homogenizing, and splitting may be performed by a field
971 laboratory prior to sample shipment to the analytical laboratory.

972 If volatile elements are among other non-volatile contaminants, samples must be fractionated
973 before drying to avoid loss of the contaminant of interest. Dried samples are homogenized by
974 mortar and pestle, jaw crusher, ball mill, parallel plate grinder, blender, or a combination of these
975 techniques and sieved to obtain a uniform sample. Sieve sizes from 35 to 200 mesh generally are
976 recommended for wet chemistry procedures. ASTM C999 correlates various mesh sizes with
977 alternative designations, inclusive of physical dimensions expressed in inches or in the metric
978 system. In addition, samples for chemical separations are usually ashed in a muffle furnace to
979 remove any remaining organic materials that may interfere with the procedures.

980 10.4.1.2 Sample Ashing

981 Soil samples that require chemical separation for radionuclide analysis may also be ashed by the
982 field laboratory. The use of the term “field laboratory” can cause confusion, since no one
983 definition is possible. It is used here to define a lab that is close to the point of sample collection.
984 In no way does it imply that there is a distinction in requirements or specifications that impact
985 quality. For soil samples, ashing is performed in a muffle furnace to remove any organic
986 materials that may interfere with radiochemical procedures.

987 **10.4.2 Sediments**

988
989 Sediments of lakes, reservoirs, cooling ponds, settling basins, and flowing bodies of surface
990 water may become contaminated as a result of direct liquid discharges, wet surface deposition, or
991 from runoffs associated with contaminated soils. Because of various chemically and physically
992 binding interactions with radionuclides, sediments serve as integrating media that are important
993 to environmental monitoring. An understanding of the behavior of radionuclides in the aquatic
994 environment is critical to designing a sampling plan, because their behavior dictates their
995 distribution and sampling locations. Sediment cores may be sampled, frozen, and then sectioned.

996 The fate of radionuclides entering surface waters and their subsequent interaction with sediment
997 is complex due to numerous mechanisms and processes that affect the initial mixing and
998 dispersion of radionuclides, their distribution in water, sediment, plants and animals, and their
999 long-term retention within these compartments. Several factors must be considered to establish
1000 appropriate sediment sampling locations and depths and are discussed briefly below.

1001 10.4.2.1 Initial Mixing and Transport Dispersion of Radionuclides Discharged to Water

1002 The rapid initial mixing phase in the nearfield is dominated by the characteristics of the effluent
1003 and the outfall structure. The extent of nearfield mixing and dilution is strongly affected by the
1004 quantity of effluent relative to the receiving body of water, the level of turbulence produced by
1005 means of the discharge momentum (jet action), the discharge buoyancy (plume action), the
1006 outfall configuration, and the depth and current flow rate in the vicinity of outfall.

1007 Predictive models have been proposed for surface and submerged discharges; single point and
1008 multi-point outfalls; deep and shallow, stagnant and flowing water; and buoyant (positive and
1009 negative) and non-buoyant effects. An understanding of the basic hydrodynamic variables that
1010 define each of these conditions will aid in the selection of sampling locations.

1011 In the case of small and medium bodies of surface waters, where vertical thermal stratification is
1012 the primary factor that determines inflow and outflow dynamics, a simple one- or two-
1013 dimensional model may be appropriate as discussed in Regulatory Guide 1.113 (NRC 1977). For
1014 large bodies of surface water where neither horizontal nor vertical homogeneity can be assumed,

1015 more complex three-dimensional dispersion models must be applied to properly assess
1016 hydrodynamics and the distribution of radionuclides in sediment. A review of numerical
1017 hydrodynamic models for large bodies of surface waters has been presented by Johnson (1980).

1018 10.4.2.2 Sediment Effect

1019 Following initial mixing in the nearfield (i.e., outfall), subsequent transport and distribution of a
1020 dissolved radionuclide is greatly impacted if the radionuclide is absorbed strongly from solution
1021 onto sediments by processes that include ion exchange, precipitation-mineral formation,
1022 complexation-hydrolysis, and oxidation-reduction. Both suspended and less-mobile bed
1023 sediments may absorb radionuclides, but suspended sediments usually absorb more efficiently
1024 per unit weight than bed sediments (Friend et al., 1965; Parker et al., 1965).

1025 The impacts of sediment absorption in a flowing body of water are obvious: the required time for
1026 sediment absorption allows the dissolved radionuclide to move considerable distances
1027 downstream before being absorbed, and sediment absorption steadily reduces the concentration
1028 of dissolved radionuclides with the result that an activity gradient is established in downstream
1029 water, sediment, and aquatic biota. Concentration gradients are further complicated by the high
1030 mobility of suspended sediments, the slow but steady erosion of bed sediments, the mobility and
1031 transfer of the radionuclide contaminant that has entered the aquatic food web, and the various
1032 mechanisms that modify sediment adsorption and desorption.

1033 10.4.2.3 Sample Preparation/Preservation

1034 In most cases, sediment is separated from water by simple decanting, but samples also may be
1035 obtained by filtering a slurry or through passive evaporation. As noted previously, care must be
1036 taken to avoid cross contamination from sampling by decontaminating or replacing tools and also
1037 from avoiding contact between successive samples. Suitable sample containers include glass or
1038 plastic jars with screw caps. The presence of volatile or semi-volatile organic and micro-
1039 organisms may impact the radionuclide concentration, therefore, samples should be kept on ice
1040 while in the field and refrigerated while awaiting radioanalysis.

1041 **10.4.3 Other Solids**

1042 1043 10.4.3.1 Structural Materials

1044 In some cases, a project plan requires sample analysis of structural materials such as concrete or
1045 steel. Concrete from floors, walls, sidewalks or road surfaces is typically collected by scabbling,
1046 coring, drilling, or chiseling. Depending on the radionuclides of interest and detection methods,
1047 these sample preparations may require crushing, pulverization, and sieving.

1048 Metal associated with structures (e.g., I-beams, rebar) or machines may be contaminated on
1049 exterior or interior surfaces or through activation may become volumetrically contaminated.
1050 Surface contamination may be assessed by swipe samples that provide a measure of removable
1051 contamination (Section 10.7) or by scraping, sandblasting, or other abrasive techniques.
1052 Volumetric contamination is frequently assessed by non-destructive field measurements that rely
1053 on gamma-emitting activation products. However, drill-shavings or pieces cut by means of a
1054 plasma arc torch may be collected for further analysis in a laboratory where they can be analyzed
1055 in a low-background environment. In general, these materials require no preservation but, based
1056 on activity/dose rate levels and sample size and weight, may require proper shielding, engineered
1057 packaging, and shipping by a licensed carrier.

1058 10.4.3.2 Biota: Samples of Plant and Animal Products

1059 The release of radionuclides to the environment from normal facility operations or as the result of
1060 an accident requires the sampling of a wide variety of terrestrial and aquatic biota. Guidance
1061 provided below is directed principally to those responsible for designing a sampling plan, who
1062 must make decisions pertaining to the type of samples that should be collected, where and how to
1063 collect the samples, and the preferred methods for sample preparation. For most biota, sample
1064 preservation usually is achieved by icing samples in the field and refrigeration until receipt by the
1065 analytical laboratory.

1066 The specific media that fall under this general category include food, domestic animals (meat and
1067 poultry), animal products, game animals, game birds, etc. The field sampling plan should
1068 describe the type of processing and preservation required.

1069 Samples of food and certain terrestrial animals are of greatest importance in environmental
1070 surveillance because they provide the most direct basis for assessing the radiation dose to man.
1071 The principal pathways for radionuclide contamination of food and plants are atmospheric
1072 deposition from airborne releases and crop irrigation from rivers, ponds or lakes receiving liquid
1073 effluents. Care should also be taken not to select a sampling site that has been fertilized or has
1074 been contaminated by runoffs from fertilized soil due to enhanced natural radioactivity content of
1075 many fertilizers (ASTM C998).

1076 To determine the dose to a population, pathway analysis may require sampling of food and biota.
1077 One example is the analysis of meat from domestic or game animals. Samples from food and
1078 biota also may be used to determine radionuclide accumulation in the environment. For example,
1079 the analysis of growth rings from trees may indicate when a radionuclide was released into the
1080 environment.

1081 Animal feeds also provide important data for determining radionuclide concentrations in the food
1082 chain. Foods may be categorized according to the U.S. Department of Agriculture scheme as
1083 leafy vegetables, grains, tree-grown fruits, etc., and representative samples from each group may

1084 be selected for analysis. Guidance for procuring or preparing terrestrial samples is provided
1085 below.

1086 MEAT, PRODUCE, AND DAIRY PRODUCTS

1087 Meat, poultry, eggs, fresh produce, and other food should be procured from local farmers most
1088 likely to have been affected by a singular event. The choice of sample is dependent on the
1089 pathway. Meat samples also may be collected at a slaughter house if the origin of the animals can
1090 be documented. Local health departments may be able to assist in getting samples. Samples
1091 should be placed in sealed plastic bags and appropriately labeled and preserved by means of ice
1092 in the field and refrigeration during interim storage prior to delivery to the analytical laboratory.
1093 All food samples may be reduced to edible portions (depending on study objective) for analysis
1094 in a manner similar to that for human consumption (i.e., remove cores, bones, seeds, other
1095 nonedible parts) and weighed as received from the field (i.e., wet weight) within 24 hours. Wet
1096 weights are desired, since consumption data are generally on this basis.

1097 For sampling fresh produce, fruits, meats, and other domestic animal products, a local land-use
1098 study may be necessary to determine what crops and animals are important in the local diet and
1099 where they are produced with respect to the site. Fruit and vegetable samples should be collected
1100 near the point of maximum predicted annual ground concentration from airborne releases and
1101 from areas that may be contaminated by water into which liquid plant wastes have been
1102 discharged (e.g., irrigated crops). Local land usage should be reviewed periodically, as well as
1103 current farming and stock-feeding practices at sampling locations.

1104 ANIMAL FEED AND VEGETATION

1105 Crops raised for animal feed and vegetation consumed by grazing farm animals may be sampled.
1106 Depending upon radionuclides under investigation and their analytical sensitivities, kilogram
1107 quantities of vegetative matter may be needed. The choice of species and sample type must be
1108 guided by factors such as exposure pathways, species availability, seasonal growth patterns, soil
1109 types, and farming practices.

1110 As in all terrestrial samples, naturally occurring ^{40}K and the uranium and thorium series
1111 contribute to the radiation observed. Deposition of such cosmic-ray-produced nuclides as ^7Be and
1112 fallout from nuclear tests also may be present. Properly selected processed items from commer-
1113 cial sources may be helpful in providing natural and anthropogenic background data.

1114 WILDLIFE

1115 Wild animals that are hunted and eaten may be of interest for potential dose estimates and
1116 therefore may require sampling. However, the data from small numbers of samples of wild
1117 animals or game birds should be viewed with caution because of their great variation in mobility,

1118 age, and diet. Examples of wildlife that have been used are rabbits and rodents that may feed on
1119 and live in a contaminated site.

1120 Wildlife samples can be trapped, acquired from hunters, collected after accidental road kills, or
1121 obtained by request to the appropriate state game agency. Wildlife that is relatively rare locally
1122 should not be taken as environmental samples. Since the choice of species samples may be
1123 crucial to the usefulness of the results, local ecologists and biologists should be consulted to
1124 ensure consideration of factors that affect animal radionuclide uptake and retention, such as size,
1125 age, sex, feeding locus, and food consumption. An estimate of the radionuclide intake of the
1126 animal just before its death may be provided by analyzing the stomach content, especially the
1127 rumen in deer. However, the sample must be collected within a brief period (two to four hours)
1128 after death.

1129 AQUATIC ENVIRONMENTAL SAMPLES

1130 In addition to natural radionuclides and natural radionuclides enhanced by human activity, there
1131 are numerous man-made radionuclides that have the potential for contaminating surface and
1132 ground water. The most common of these are fission and activation products associated with
1133 reactor operation and fuel cycle facilities. Radioanalysis of aquatic samples may therefore
1134 include ^{54}Mn , ^{58}Co , ^{60}Co , ^{65}Zn , ^{95}Zr , ^{90}Sr , ^{134}Cs , ^{137}Cs , and transuranics, such as ^{239}Pu .

1135 When surface and ground waters are contaminated, radionuclides may be transferred through a
1136 complex food web consisting of aquatic plants and animals. Aquatic plants and animals, as
1137 discussed here, are any species which derive all or substantial portions of their nourishment from
1138 the aquatic ecosystem, are part of the human food chain, and show significant accumulation of a
1139 radionuclide relative to its concentration in water. Although fish, aquatic mammals, and
1140 waterfowl provide a direct link to human exposure, lower members of the food chain also may be
1141 sampled.

1142 FLORA

1143 Aquatic biota such as algae, seaweed, and benthic organisms are indicators and concentrators of
1144 radionuclides—especially ^{59}Fe , ^{60}Co , ^{65}Zn , ^{90}Sr , and ^{137}Cs —and can be vectors in the water-fish-
1145 human food chain. As such, they may be sampled upstream and downstream at locations similar
1146 to those described for sediment. Because of their high water content, several kilograms (wet
1147 weight) should be collected per sample. The wet weight of the sample should be recorded.
1148 Enough of the wet sample should be processed so that sufficient sample remains following the
1149 drying process. Both algae (obtained by filtering water or by scraping submerged substrates) and
1150 rooted aquatic plants should be sampled.

1151 FISH AND SHELLFISH

1152 For practical reasons, fish and shellfish may be purchased from local sources if the origin can be
1153 determined. Samples also can be obtained by pole fishing, netting, or electric shock devices. The
1154 sampling plan will describe the processing needed. Samples should include each of the principal
1155 edible types in local catches. Several kilograms of each fish sample are usually required; this may
1156 be one large fish, but preferably a composite of a number of small ones. Analysis of the edible
1157 portions of food fish as prepared for human consumption is of major interest. Fish may be de-
1158 boned, if specified in the sampling plan. The whole fish is analyzed if it is used for the
1159 preparation of a fish meal for consumption or if only trend indication is required. In a program
1160 where fish are the critical pathway, fish are analyzed by species; if less detail is required, several
1161 species with similar feeding habits (such as bottom feeders, insectivores, or predators) may be
1162 collected and the data grouped.

1163 In large bodies of water, samples from several locations are desirable because of the difficulty in
1164 knowing whether a fish caught at a given location had lived there for an extended period. Thus,
1165 the presence or absence of a radionuclide in a specific fish does not permit any definite
1166 conclusion concerning the presence of the radionuclide in water at that location. For some fish,
1167 more specific information concerning their usual location may be available; for example, dams,
1168 salinity gradients, and temperature gradients can be effective barriers to their movement.
1169 Information on fish age, feeding habits, and the quality of the aquatic environment are desirable
1170 to evaluate the significance of any findings.

1171 Shellfish, such as clams, oysters, and crabs, are collected for the same reasons as fish, but have
1172 the advantage as indicators of being relatively stationary. Their restricted mobility contributes
1173 substantially to the interpretation and application of analytical results to environmental
1174 surveillance. Edible and inedible portions of these organisms can be prepared separately.

1175 WATERFOWL

1176 Waterfowl, such as ducks and geese, may also concentrate radionuclides from their food sources
1177 in the aquatic environment and serve as important food sources to humans. The migratory
1178 patterns and feeding habits of waterfowl vary widely. Some species are bottom feeders and, as
1179 such, tend to concentrate those radionuclides associated with sediments such as ⁶⁰Co, ⁶⁵Zn, and
1180 ¹³⁷Cs. Others feed predominantly on surface plants, insects, or fish.

1181 Whenever practical, and if time permits, waterfowl should be obtained by hunting, but a trapping
1182 procedure may also be used. An important consideration in obtaining a sample from waterfowl is
1183 that their exterior surfaces, especially feathers, may be contaminated. It is important to avoid
1184 contaminating the “flesh” sample during handling. As with other biota samples, analyses may be
1185 limited to the edible portions and should be reported on a wet weight basis. Local game officials
1186 or aquatic ecologists may provide valuable information for choosing the proper species.

1187 Caution is advised in the selection of background or control locations for all biota (terrestrial and
1188 aquatic) sampled, at least for those species whose mobility and feeding habits may significantly
1189 affect the results obtained. Since this mobility makes it difficult to establish upstream/
1190 downstream sampling locations for biota in a manner analogous to those for air, water, or plants,
1191 a sound sampling strategy may require the expert advice and direction of local ecologists, and
1192 fish and game personnel. Samples from the background locations should be from an ecosystem
1193 identical to that of those collected near the site, but unaffected by site effluents.

1194 **10.5 Air Sampling**

1195 The measurement of airborne radionuclides as gases or particulates provides a means of
1196 evaluating internal exposure through the inhalation pathways. The types of airborne radioactivity
1197 that may require air sampling are normally categorized as: (1) airborne particulates; (2) noble
1198 gases; (3) volatilized halogens (principally radioiodines); and (4) tritiated water. Depending upon
1199 the source term and the objectives of the investigation, air sampling may be conducted outdoors
1200 as well as indoors on behalf of a variety of human receptors. For example, routine outdoor air
1201 samples may be taken for large population groups living within a specified radius of a nuclear
1202 facility. On the other end of the spectrum, air samples may be taken for a single person or small
1203 group of persons exposed occupationally to a highly localized source of airborne radioactivity.

1204 The purpose of the samples being collected must, therefore, be well defined in terms of sampling
1205 location, field sampling equipment, and required sample volumes. Due to the wide range of
1206 conditions that may mandate air sampling, and the limited scope of this section, only generic
1207 topics of air sampling will be discussed.

1208 **10.5.1 Sampler Components**

1209 Common components of air sampling equipment include a sample collector (i.e., filter), a sample
1210 collector holder, an air mover, and a flow-rate measuring device.

1211 The sample holder should provide adequate structural support while not damaging the filter,
1212 should prevent sampled air from bypassing the filter, should facilitate changing the filter, and
1213 should facilitate decontamination. A backup support that produces negligible pressure drop
1214 should be used behind the filter to prevent filter distortion or deterioration.

1215 If rubber gaskets are used to seal the filter to the backing plate, the gasket should be in contact
1216 with the filter along the entire circumference to ensure a good fit.

1217 Air movers or vacuum systems should provide the required flow through the filter and to
1218 minimize air flow reduction due to filter loading. Consideration should be given to the use of air
1219 movers that compensate for pressure drop. Other factors to consider should include size, power
1220 consumption, noise, durability, and maintenance requirements.

1221 Each air sampler should be equipped with a reliable calibrated air flow measuring device with
1222 specified accuracy. To calculate the concentrations of any radionuclide in air collected, it is
1223 necessary to accurately determine the total volume of air sampled. The planning documents
1224 should state who is responsible for making volume corrections. Also, the information needed for
1225 half-life corrections for short-lived radionuclides needs to be recorded.

1226 Generally, a parameter of the air mover can be related to flow. If the mean flow during a
1227 collection period can be determined, the total volume of air sampled can be readily calculated.
1228 Accurate flow measurements and the total integrated sample volume of air can be obtained using
1229 a mass flow meter and a totalizer. This direct technique of air flow measurement becomes
1230 impractical at remote field locations, due to cost and exposure of the flow meter to harsh
1231 environments. Other procedures for the measurement of air flow in sampling systems are
1232 reviewed by Lippmann (1989a). The equipment readings (flow rate, volume, etc.) should be
1233 recorded by the sample collector.

1234 The collection medium or filter used depends on the physical and chemical properties of the
1235 materials to be collected and counted. A variety of particulate filters (cellulose, cellulose-
1236 asbestos, glass fiber, membrane, polypropylene, etc.) is available. The type of filter is selected
1237 according to needs, such as high collection efficiency, particle-size selectivity, retention of alpha
1238 emitters on the filter surface, and the compatibility with radiochemical analysis. The criteria for
1239 filter selection are good collection efficiency for submicron particles at the range of face
1240 velocities used, high particle and mass loading capacity, low-flow resistance, low cost, high
1241 mechanical strength, low-background activity, compressibility, low-ash content, solubility in
1242 organic solvents, non-hygroscopicity, temperature stability, and availability in a variety of sizes
1243 and in large quantities. The manufacturer's specifications and literature should provide a source
1244 for filter collection efficiency. In the selection of a filter material, a compromise must be made
1245 among the above-cited criteria that best satisfies the sampling requirements. An excellent review
1246 of air filter material used to monitor radioactivity was published by Lockhart and Anderson
1247 (1964). Lippmann (1989b) also provides information on the selection of filter materials for
1248 sampling aerosols by filtration. See ANSI (1999), Annex D and Table D.1, for criteria for the
1249 selection of filters for sampling airborne radioactive particles.

1250 In order to select a filter medium with adequate collection efficiency, it may be necessary to first
1251 determine the distribution of size of airborne particulates. Several methods, including impactors
1252 (e.g., multistage cascade impactor) and electrostatic precipitators, can be used to classify particle
1253 size. Waite and Nees (1973) and Kotrappa et al. (1974) discuss techniques for particle sizing
1254 based on the flow discharge perturbation method and the HASL cyclone, respectively. These
1255 techniques are not recommended for routine environmental surveillance of airborne particulates,
1256 although their use for special studies or for the evaluation of effluent releases should not be
1257 overlooked. Specific data on various filter materials, especially retention efficiencies, have been
1258 reported by several authors (Lockhart and Anderson, 1964; Denham, 1972; Stafford, 1973;
1259 ASTM STP555) and additional information is available from manufacturers.

1260 **10.5.2 Filter Selection Based on Destructive Versus Non-destructive Analysis**

1261 Pure cellulose papers are useful for samples to be dissolved and analyzed radiochemically, but
1262 the analytical filter papers used to filter solutions are inefficient collectors for aerosols and clog
1263 easily. Cellulose-asbestos filter papers combine fairly high efficiency, high flow rates, high
1264 mechanical strength, and low pressure drops when loaded. They are very useful for collecting
1265 large samples but present difficulties in dissolution, and their manufacture is diminishing because
1266 of the asbestos. Fiberglass filters can function efficiently at high flow rates, but require fluoride
1267 treatment for dissolution and generally contain sufficient radioactive nuclides to complicate low-
1268 activity analysis. Polystyrene filters are efficient and capable of sustaining high air flow rates
1269 without clogging. They are readily destroyed for analysis by ignition (300° C) or by wet washing
1270 with oxidizing agents, and also are soluble in many organic liquids. They have the disadvantage
1271 of low mechanical and tensile strength, and they must be handled carefully. Membrane filters are
1272 excellent for surface collection efficiency and can be used for direct alpha spectrometry on the
1273 filter. However, they are fragile and suffer from environmental dust loading. An alternative
1274 choice for radionuclides in the environment is the polypropylene fiber filter, Dynaweb Grade
1275 DW7301L. Filters come in two sizes: a 20.32 cm circle and a 20.32 cm x 25.40 cm rectangle.
1276 The filter is composed of a 100 percent polypropylene web that is 100 percent binderless. Three
1277 layers of this web are collated and sandwiched between two sheets of a protective DuPont Reeme
1278 (100 percent polyester) scrim.

1279 **10.5.3 Sample Preservation and Storage**

1280 Since particulate air samples are generally dry samples that are chemically and physically stable,
1281 they require no preservation. However, care must be exercised to avoid loss of sample from the
1282 filter medium and the cross contamination among individual samples. A common method is to
1283 fold filters symmetrically so that the two halves of the collection surface are in contact. Filters
1284 should be stored in individual envelopes that have been properly labeled. Filters may also be
1285 stored in special holders that attach on the filter's edge outside of the collection surface.
1286 When background levels of ²²²Ra and ²²⁰Ra progeny interfere with evaluation of alpha air
1287 samples, a holdup time of several hours may be required before samples are counted. Corrections
1288 or determinations can also be made for the contribution of radon or thoron progeny present on a
1289 filter (Setter and Coats, 1961).

1290 **10.5.4 Special Cases: Collection of Gaseous and Volatile Air Contaminants**

1291 Prominent radionuclides that may exist in gaseous states include noble gases, ¹⁴C as carbon
1292 dioxide or methane, ³H as water vapor, and volatilized radioiodines. (Radon is discussed in
1293 Section 10.5.5.)

1294 10.5.4.1 Radioiodines

1295 The monitoring of airborne iodine, such as ¹²⁹I and ¹³¹I, may be complicated by the probable
1296 existence of several species, including particulate iodine or iodine bound to foreign particles,
1297 gaseous elemental iodine, and gaseous non-elemental compounds of iodine. A well-designed
1298 sampling program should be capable of distinguishing all possible iodine forms. While it may
1299 not always be necessary to differentiate between the various species, care should be taken so that
1300 no bias can result by missing one or more of the possible species. See ANSI (1999) Annex C.3,
1301 for information on collection media for radioiodine.

1302 In addition to the problems noted above, charcoal cartridges (canisters) for the collection of
1303 radioiodine in air are subject to channeling. Hence, they should be carefully checked before
1304 operation in the field (analogous to DOP testing of high efficiency particulate air (HEPA) filters
1305 *in situ*) or several should be mounted in series to prevent loss of iodine. Too high a sampling rate
1306 reduces both the collection efficiency and retention time of charcoal filters, especially for the
1307 non-elemental forms of iodine (Keller et al., 1973; Bellamy, 1974). The retention of iodine in
1308 charcoal is dependent not only on charcoal volume, but also the length of the charcoal bed.
1309 Typical air flow rates for particulate sampling of 30 to 90 L/min (1 to 3 ft³/min) are normally
1310 acceptable for environmental concentrations of radioiodine. The method proposed by the
1311 Intersociety Committee (APHA, 1972) for ¹³¹I concentrations in the atmosphere involves
1312 collecting iodine in its solid and gaseous states with an “absolute” particulate filter in series with
1313 an activated charcoal cartridge followed by gamma spectrometric analysis of the filter and
1314 cartridge. The Intersociety-recommended charcoal cartridges are 5/8 in. diameter by 1.5 in. deep
1315 containing 3 g of 12 to 30 mesh KI-activated charcoal. The minimum detectable level using the
1316 Intersociety method is 3.7×10^{-3} Bq/m³ (0.1 pCi/m³). Larger cartridges will improve retention,
1317 permitting longer sampling periods. A more sensitive system has been described by Baratta et al.
1318 (1968), in which concentrations as low as 0.037 Bq/m³ (0.01 pCi/mL) of air are attainable.

1319 For the short-lived radioiodines (mass numbers 132, 133, 135), environmental sampling is
1320 complicated by the need to obtain a sufficient volume for analysis, while at the same time,
1321 retrieving the sample soon enough to minimize decay (with half-lives ranging from two hours to
1322 31 hours). Short period (grab) sampling with charcoal cartridges is possible, with direct counting
1323 of the charcoal as soon as possible for gamma emissions, but radon and thoron will affect
1324 detection levels.

1325 Because of the extremely long half-life and normally low environmental concentrations, ¹²⁹I
1326 determinations must usually be performed by neutron activation or mass spectrometry analysis
1327 after chemical isolation of the iodine. For concentrations about 3×10^{-10} μCi/mL, liquid
1328 scintillation counting can be used after solvent extraction (Gabay et al., 1974).

1329 10.5.4.2 Gases

1330 Sampling for radioactive gases is either done by grab sample that employs an evacuated chamber
1331 or by airflow through a medium such as charcoal, water, or a variety of chemical absorbers. For
1332 example, radioactive CO₂ is most commonly extracted by passing a known volume of air through
1333 columns filled with 3 M NaOH solution. After the NaOH is neutralized with sulfuric acid, the
1334 CO₂ is precipitated in the form of BaCO₃, which then can be analyzed in a liquid scintillation
1335 counter (NCRP,1985).

1336 Because noble gases have no metabolic significance, and concern is principally limited to
1337 external exposure, surveillance for noble gases is commonly performed by ambient dose rate
1338 measurements. However, the noble gases xenon and krypton may be extracted from air by
1339 adsorption on activated charcoal (Scarpitta and Harley, 1990). However, depending upon the
1340 analytical method and instrumentation employed, significant interference may result from the
1341 presence of naturally occurring radioactive gases of ²²²Rn and ²²⁰Rn.

1342 10.5.4.3 Tritium Air Sampling

1343 In air, tritium occurs primarily in two forms: as water vapor (HTO) and as hydrogen gas (HT).
1344 Tritiated organic compounds in the vapor phase or attached to particulate matter occur only
1345 occasionally. To measure tritium as HT or in tritiated organic, the gas phase can be oxidized,
1346 converting the tritium to HTO before desiccation and counting. For dosimetric purposes, the
1347 fraction present as HT can usually be neglected, since the relative dose for a given activity
1348 concentration of HTO is 400 times that for HT (NCRP, 1978). However, if HT analysis is
1349 required, it can be removed from the atmosphere by oxidation to water (HTO) using CuO/MnO₂
1350 at 600° C (Pelto et al., 1975), or with air passed over platinum alumina catalyst (Bixel and
1351 Kershner 1974). These methods also oxidize volatile tritiated organic compounds to yield
1352 tritiated water (ANSI, 1999, Annex H).

1353 A basic system for sampling HTO consists of a pump, a sample collector, and a flow-measuring
1354 or flow-recording device. Air is drawn through the collector for a measured time period at a
1355 monitored flow rate to determine the total volume of air sampled. The total amount of HTO
1356 recovered from the collector is divided by the total volume of air sampled to determine the
1357 average HTO-in-air concentration of the air sampled. In some sampler types, the specific activity
1358 of the water collected is measured and the air concentration is determined from the known or
1359 measured humidity. Some common collectors are cold traps, tritium-free water, and solid
1360 desiccants, such as silica gel, DRIERITE™, or molecular sieve.

1361 Cold traps are usually made of glass and consist of cooled collection traps through which sample
1362 air flows. The trap is cooled well below the freezing point of water, usually with liquid nitrogen.
1363 The water vapor collected is then prepared for analysis, usually by liquid scintillation counting.
1364 Phillips and Easterly (1982) have shown that more than 95 percent HTO collection efficiency can

1365 be obtained using a single cold trap. Often a pair of cold traps is used in series, resulting in a
1366 collection efficiency in excess of 99 percent.

1367 Gas-washing bottles (i.e., “bubblers”) filled with an appropriate collecting liquid (usually tritium-
1368 free water) are used quite extensively for collecting HTO from air. HTO in the sample gas stream
1369 “dissolves” in the collecting liquid. For the effective collection rate to remain the same as the
1370 sample flow rate, the specific activity of the bubbler water must be negligible with respect to the
1371 specific activity of the water vapor. Thus, the volume of air that can be sampled is ultimately
1372 limited by the volume of water in the bubbler. However, except when sampling under conditions
1373 of very high humidity, sample loss (dryout) from the bubbler usually limits collection time rather
1374 than the attainment of specific activity equilibrium. Osborne (1973) carried out a thorough
1375 theoretical and experimental evaluation of the HTO collection efficiency of water bubblers over a
1376 wide range of conditions.

1377 The use of silica gel as a desiccant to remove moisture from air is a common technique for
1378 extracting HTO. The advantage of using silica gel is that lower HTO-in-air concentrations can be
1379 measured, since the sample to be analyzed is not significantly diluted by an initial water volume,
1380 which occurs when a liquid-sampling sink is used. Correcting for dilution is discussed in Rosson
1381 et al. (2000).

1382 **10.5.5 Radon**

1383 There are three isotopes of radon in nature: ^{222}Rn is a member of the ^{238}U decay chain; ^{220}Rn is a
1384 member of the ^{232}Th decay chain; and ^{219}Rn is a member of the ^{235}U decay chain. Because of the
1385 small relative abundance of the parent nuclides and the short half-lives of ^{220}Rn (55 seconds) and
1386 ^{219}Rn (4 seconds), the term “radon” generally refers to the isotope ^{222}Rn . Owing to its ubiquitous
1387 presence in soils, uranium mill tailings, underground mines, etc., and the health risks to large
1388 populations and occupational groups, radon is perhaps the most studied radionuclide.

1389 Consequently, many reports and articles have been published in the scientific literature dealing
1390 with the detection methods and health risks from radon exposures. Many of them appear in
1391 publications issued by the EPA, DOE, NCRP, NAS, and in radiation-related journals, such as the
1392 journals *Health Physics* and *Radiation Research*. Given the voluminous amount of existing
1393 information, only a brief overview of the sampling method can be presented here.

1394 10.5.5.1 Radon Sampling Methods

1395 Quantitative measurements of radon gas and its short-lived decay products can be obtained by
1396 several techniques that are broadly categorized as grab sampling, continuous radon monitoring,
1397 and integrative sampling. Each method imposes unique requirements that should be followed
1398 carefully. The U.S. EPA Radon Measurement Proficiency (RMP) Program should be consulted
1399 for current guidance for sample collection (EPA, 1992; EPA, 1993). Information is available on

1400 the RMP home page at www.epa.gov/radonpro/index.htm. Working with the Radon Proficiency
1401 Program (RPP) is described in a separate handbook (EPA, 1996). A description of additional
1402 sampling methods and materials is also presented in EPA (1994) and Cohen (1989).

1403 In general, EPA's protocols specify that radon sampling and measurements be made under
1404 standardized conditions when radon and its progeny are likely to be at their highest concentra-
1405 tions and maximum equilibrium. For indoor radon measurement, this implies minimum building
1406 ventilation through restrictions on doors, windows, HVAC systems, etc. Also sampling should
1407 not take place during radical changes in weather conditions. Both high winds and rapid changes
1408 in barometric pressure can dramatically alter a building's natural ventilation rate. Although
1409 recommended measurements are likely to generate higher than actual average concentrations, the
1410 benefit of a standardized sampling condition is that it is reproducible, least variable, and
1411 moderately conservative. Brief descriptions of the basic techniques used to sample air for radon
1412 and its progeny are provided below.

1413 GRAB SAMPLING

1414 The term "grab sampling" refers to very short-term sampling. This method consists of evaluating
1415 a small volume of indoor air for either radon or radon decay product concentration. In the radon
1416 grab sampling method, a sample of air is drawn into and subsequently sealed in a flask or cell
1417 that has a zinc sulfide phosphor coating on its interior surfaces. One surface of the cell is fitted
1418 with a clear window that is put in contact with a photomultiplier tube to count light pulses
1419 (scintillations) caused by alpha disintegrations from the sample interacting with the zinc sulfide
1420 coating. The number of pulses is proportional to the radon concentration in the cell. The cell is
1421 counted about four hours after filling to allow the short-lived radon decay products to reach
1422 equilibrium with the radon. The results are corrected to compensate for decay during the time
1423 between collection and counting, and for decay during counting.

1424 Several methods for performing such measurements have been developed. However, two
1425 procedures that have been most widely used with good results are the Kusnetz procedure and the
1426 modified Tsivogiou procedure. In brief, the Kusnetz procedure (Kusnetz, 1956; ANSI, 1973)
1427 may be used to obtain results in working levels (WL) when the concentration of individual decay
1428 products is not important. Decay products in up to 100 liters of air are collected on a filter in a
1429 five-minute sampling period. The total alpha activity on the filter is counted any time between 40
1430 and 90 minutes after sampling is completed. Counting can be done using a scintillation-type
1431 counter to obtain gross alpha counts for a selected counting time. Counts from the filter are
1432 converted to disintegrations using the appropriate counter efficiency. The disintegrations from
1433 the decay products may be converted into working levels using the appropriate "Kusnetz factor"
1434 for the counting time used.

1435 The Tsivogiou procedure may be used to determine both WL and the concentration of the
1436 individual radon decay products. Sampling is the same as in the Kusnetz procedure. However,

1437 the filter is counted three separate times following collection. The filter is counted between 2 and
1438 5 minutes, 6 and 20 minutes, and 21 and 30 minutes after sampling is complete. Count results are
1439 interpreted by a series of equations that calculate concentrations of the three radon decay
1440 products and WL.

1441 The advantages of grab sampling are that the analysis time is relatively short, results are available
1442 within a short time, and conditions during the measurement are known to the sampler. In
1443 addition, grab sampling does not provide a long-term average and house conditions must be
1444 controlled for 12 hours prior to measurement.

1445 CONTINUOUS RADON MONITOR

1446 A continuous radon monitor (CRM) samples the ambient air by pumping air into a scintillation
1447 cell after passing it through a particulate filter that removes dust and radon decay products. As
1448 the radon in the air decays, the ionized radon decay products plate out on the interior surface of
1449 the scintillation cell. As the radon decays, the alpha particles strike the coating on the inside of
1450 the cell, causing scintillations. The scintillations are detected by the photomultiplier tube in the
1451 detector, which generates electrical signals. The signals are processed and the results are either
1452 stored in the memory of the CRM or printed on paper tape by the printer. The CRM must be
1453 calibrated in a known environment to obtain the conversion factor used to convert count to radon
1454 concentration.

1455 The CRM may be a flowthrough-cell type or a periodic-fill type. In the flowthrough-cell type, air
1456 flows continuously into and through the scintillation cell. The periodic-fill type fills the cell once
1457 during each preselected time interval, counts the scintillations, then begins the cycle again.

1458 An analogous device to the continuous radon monitor is the Continuous Working Level Monitor
1459 (CWLM). This device filters air at a low flow rate of about 0.2 to one liter per minute and
1460 measures the amount of radon decay products on the filter medium. An alpha detector, such as a
1461 diffused-junction or surface-barrier detector, counts the alpha particles produced by the radon
1462 decay products as they decay on the filter. The detector is normally set to detect alpha particles
1463 with energies between 2 and 8 meV. The alpha particles emitted from the radon decay products
1464 ^{218}Po and ^{214}Po are the significant contributors to the events that are measured by the detector.
1465 The event count is directly proportional to the number of alpha particles emitted by the radon
1466 decay products on the filter. The unit typically contains a microprocessor that stores the number
1467 of counts and elapsed time. The unit can be set to record the total counts registered over specified
1468 time periods. The unit must be calibrated in a calibration facility to convert count rate to working
1469 level (WL) values. This may be done initially by the manufacturer and should be done
1470 periodically thereafter by the operator.

1471 INTEGRATING SAMPLING DEVICES

1472 By far, the most common technique for measuring radon is by means of integrating devices.
1473 Integrating devices, like the charcoal canister and the Electret-Passive Environmental Radon
1474 Monitor, are commonly employed as short-term integrating devices (two to seven days), while
1475 alpha track detectors are commonly used to provide measurements of average radon levels over
1476 periods of weeks to months.

1477 CHARCOAL CANISTERS

1478 Charcoal canisters (CC) are passive devices requiring no power to function. The passive nature
1479 of the activated charcoal allows continual adsorption and desorption of radon. During the
1480 measurement period, the adsorbed radon undergoes radioactive decay. Therefore, the technique
1481 does not uniformly integrate radon concentrations during the exposure period. As with all
1482 devices that store radon, the average concentration calculated using the mid-exposure time is
1483 subject to error if the ambient radon concentration adsorbed during the first half of the sampling
1484 period is substantially higher or lower than the average over the period. For a 2 to 7 day exposure
1485 period, the minimum detectable concentration (MDC) should be 18.5 Bq/m³ (0.5 pCi/L) or less
1486 (EPA, 1989). This detection level can normally be achieved with a counting time of up to 30
1487 minutes. This MDC should be calculated using the results of charcoal background
1488 determinations. The coefficient of variation should not exceed 10 percent (1 sigma) at radon
1489 concentrations of 148 Bq/m³ (4 pCi/L) or greater (EPA, 1989). This precision should be
1490 monitored using the results of duplicate canister analyses. CCs can achieve an average coefficient
1491 of variation of less than five percent at concentrations of 148 Bq/m³ (4 pCi/L) or greater.

1492 ELECTRET-PASSIVE ENVIRONMENTAL RADON MONITORS

1493 Electret-passive environmental radon monitors (E-perms) require no power and function as true
1494 integrating detectors that measure the average concentration during the exposure period. E-
1495 PERMS contain a permanently charged Electret (an electrostatically charged disk of Teflon) that
1496 collects ions formed in the chamber by radiation emitted from radon decay products. When the
1497 device is exposed, radon diffuses into the chamber through filtered openings. Ions that are
1498 generated continuously by the decay of radon and radon decay products are drawn to the surface
1499 of the electret and reduce its surface voltage. The amount of voltage reduction is related directly
1500 to the average radon concentration present during the exposure period. There are both short-term
1501 (2 to 7 days) and long-term (1 to 12 months) E-PERMS that are marketed currently. The
1502 thickness of the electret affects the usable measurement period. For a 7-day exposure period
1503 using a short-term E-PERM, as well as for a long-term E-PERM, the MDC is about 11.1 Bq/m³
1504 (0.3 pCi/L) (EPA, 1989). The coefficient of variation should not exceed 10 percent (1 sigma) at
1505 radon concentrations of 148 Bq/m³ (4 pCi/L) or greater. This precision should be verified by
1506 using results of duplicate detector analysis.

1507 ALPHA TRACK DETECTORS

1508 An alpha track detector (ATD) consists of a small piece of plastic or film enclosed in a container
1509 with a filter-covered opening. Radon diffuses through the filter into the container and alpha
1510 particles emitted by radon and its decay products strike the detector and produce submicroscopic
1511 damage tracks. At the end of the measurement period, the detectors are returned to a laboratory.
1512 Plastic detectors are placed in a caustic solution that accentuates the damage tracks so they can be
1513 counted using a microscope or an automated counting system. The number of tracks per unit area
1514 is correlated to the radon concentration in air, using a conversion factor derived from data
1515 generated at a calibration facility. The number of tracks produced per unit time is proportional to
1516 the radon concentration, so an ATD functions as a true integrating detector and measures the
1517 average concentration over the measurement period. The MDC and precision of an ATD system
1518 is dependent upon the tracks counted and, therefore, the area of the detector that is analyzed.
1519 With present ATDs, routine counting achieves a MDC of 6,660 Bq/m³-days (180 pCi/L-days).
1520 The coefficient of variation (precision) should be monitored using the results of duplicate
1521 detectors. The coefficient of variation should not exceed 20 percent (1 sigma) at radon
1522 concentrations of 148 Bq/m³ (4 pCi/L) or greater (EPA, 1989).

1523 10.5.5.2 Selecting a Radon Sampling Method Based on Data Quality Objectives

1524 The choice from among the sampling methods described above depends on whether the measure-
1525 ment is intended as a quick screening measurement or as a measurement that determines average
1526 exposure. In practice, the choice of a measurement system often is dictated by availability. If
1527 alternative systems are available, the cost or duration of the measurement may become the
1528 deciding factor. Each system has its own advantages and disadvantages, and the investigator
1529 must exercise some judgment in selecting the system best suited to the DQOs of the
1530 investigation.

1531 There are, however, some general guidelines concerning standardized measurement conditions
1532 and quality assurance objectives which apply to all measurement techniques. The following
1533 elements of quality assurance should be included in any measurement program: detector
1534 calibrations, replicate measurements, background measurements, and routine sensitivity checks.

1535 Detector calibrations are measurements made in a known radon environment, such as a
1536 calibration chamber. Detectors requiring laboratory readout, such as charcoal canisters and alpha-
1537 track detectors, should be exposed in the calibration chamber and then analyzed. Instruments
1538 providing immediate results, such as continuous working-level monitors and continuous radon
1539 monitors, should be operated in a chamber to establish calibration.

1540 There are two types of calibration measurements that should be made for alpha-track detectors
1541 and charcoal canisters. The first measurements determine and verify the conversion factors used
1542 to derive the concentration results. These measurements, commonly called spiked samples, are

1543 done at the beginning of the measurement program and periodically thereafter. The second
1544 calibration measurements monitor the accuracy of the system. These are called blind calibration
1545 measurements and consist of detectors that have been exposed in a radon calibration chamber.
1546 The detectors are not labeled as such when sent to a processing laboratory.

1547 Background measurements, or blanks, should also be conducted. Such measurements should be
1548 made using unexposed passive detectors, or should be instrument measurements conducted in
1549 very low (outdoor) radon concentration environments and separated from the operating program.
1550 Generally, these should be equivalent in frequency to the spiked samples and should also not be
1551 identified as blanks when submitted for analysis to external laboratories. In addition to these
1552 background measurements, the organization performing the measurements should calculate the
1553 minimum detectable concentration MDC for the measurement system. This MDC is based on the
1554 system's background and can restrict the ability of some measurement systems to measure low
1555 concentrations.

1556 Duplicate measurements provide an estimate of the precision of the measurement results.
1557 Duplicate measurements should be included in at least 10 percent of the samples. If enough
1558 measurements are made, the number of duplicates may be reduced, as long as enough are used to
1559 analyze the precision of the method.

1560 A quality assurance program should include a written plan for satisfying the preceding
1561 objectives. A system for monitoring the results of the four types of quality assurance
1562 measurements should also be maintained.

1563 Calibrated radon detection devices and on-site measurements can also be obtained under contract
1564 from commercial vendors who have demonstrated their proficiency in measuring radon and
1565 radon decay products, and who have had their quality assurance programs assessed by the EPA or
1566 state agencies.

1567 **10.6 Wipe Sampling for Assessing Surface Contamination**

1568 Surface contamination falls into two categories: fixed and loose. The wipe test (also referred to
1569 as “swipes” or “smears”) is the universally accepted technique for detecting removable
1570 radioactive contamination on surfaces (Section 12.5). It is often a stipulation of radioactive
1571 materials licenses and is widely used by laboratory personnel to monitor their work areas,
1572 especially for low-energy radionuclides that are otherwise difficult to detect with hand-held
1573 survey instruments. A comprehensive history of “Use of Smears for Assessing Removable
1574 Contamination” is presented by Frame and Abelquist (1999).

1575 The U.S. Nuclear Regulatory Commission (NRC, 1981) suggests that 100 cm² areas be wiped
1576 and lists acceptable levels for surface contamination. However, NRC neither recommends the
1577 collection device nor the manner in which to conduct such surveys, relying instead on

1578 suggestions by the National Committee on Radiation Protection (1964) and the National Council
1579 on Radiation Protection and Measurements (1978).

1580 **10.6.1 Sample Collection Methods**

1581 10.6.1.1 Dry Wipes

1582 Smears for removable surface activity are obtained by wiping an area of approximately 100 cm²
1583 using a dry filter paper, such as Whatman 50 or equivalent, while applying moderate pressure. A
1584 47 mm diameter filter is typically used, although for surveys for low-energy beta emitters,
1585 smaller sizes may be more appropriate because they can be placed directly into a liquid
1586 scintillation vial for counting. Small pieces of wipes occasionally are used for smears for tritium
1587 (Slobodine and Grandlund, 1974). A smear for removable contamination is obtained at each
1588 location of direct surface activity measurement.

1589 For surveys of small penetrations, such as cracks or anchor-bolt holes, cotton swabs are used to
1590 wipe the area of concern. Samples (smears or swabs) are placed into envelopes or other
1591 individual containers to prevent cross-contamination while awaiting analysis. Smears for alpha
1592 and medium- or high-energy beta activity can be evaluated in the field by counting them on an
1593 integrating scaler unit with appropriate detectors; the same detectors utilized for direct
1594 measurements may be used for this purpose. However, the more common practice is to return the
1595 smears to the laboratory, where analysis can be conducted using more sensitive techniques. The
1596 most common method for analyzing wipe samples is to use a proportional counter. For very low-
1597 energy beta emissions, wipe samples are commonly analyzed by liquid scintillation counting.

1598 10.6.1.2 Wet Wipes

1599 Although dry wipes are more convenient to handle, and there are fewer chances of cross
1600 contamination, a general limitation of dry wipes is their low recovery of surface contamination.
1601 The low recovery using dry wipes is due to the higher affinity for the surface by the contaminant
1602 than for the filter paper. Several studies have shown that for maximum sensitivity, a wipe
1603 material moistened with a suitable solvent may be indicated. For example, Ho and Shearer
1604 (1992) found that alcohol-saturated swabs were 100 times more efficient at removing
1605 radioactivity than dry swabs.

1606 In another study, Kline et al. (1992) assessed the collection efficiency of wipes from various
1607 surfaces that included vinyl floor tile, plate glass, and lead foil. Two different collection devices,
1608 cotton swabs and 2.5 cm diameter glass fiber filter disks, were evaluated under various collection
1609 conditions. Dry wipes were compared to collections made with the devices dampened with
1610 different amounts of either distilled H₂O, 70 percent ethanol, or a working-strength solution of a
1611 multipurpose laboratory detergent known to be effective for removing contaminants from
1612 laboratory glassware (Manske et al., 1990).

1613 The entire area of each square was manually wiped in a circular, inwardly-moving motion with
1614 consistent force. The collection capacity of each device was estimated by wiping progressively
1615 larger areas (multiple grids) and comparing the measured amounts of radioactivity with the
1616 amounts placed on the grids.

1617 Collection efficiency varied with both the wipe method and the surface wipe. Contamination was
1618 removed most readily from unwaxed floor tile and glass; lead foil released only about one-half
1619 the radioactivity. Stainless steel, another common laboratory surface, has contamination retention
1620 properties similar to those of glass.

1621 In most cases, collection was enhanced by at least a factor of two after dampening either the
1622 swabs or filter disks with water. Dampening with ethanol or the detergent produced removals that
1623 were statistically indistinguishable from samples dampened with an equal amount of water.

1624 The filter disks had a higher collection capacity for removable contaminants than cotton swabs,
1625 nearly doubling the radioactivity removed for each doubling of surface area wiped. Variability
1626 within all methods was high, with coefficients of variation ranging from 2 to 30 percent.

1627 For the moistened wipes, wipe efficiency depended on three factors, including the polarity of the
1628 solvent, the polarity of the contaminant being measured, and the affinity of the compound for the
1629 contaminated surface. For a solvent to readily dissolve a compound (i.e., remove it from the
1630 surface), the solvent and the compound must have similar polarities. Nonpolar solvents include
1631 ethyl acetate and petroleum ether; for polar solvents, water or methanol may be used (Cambell et
1632 al., 1993). There are other factors that influence the affinity of a compound for a surface,
1633 including porosity of the surface and available binding sites on the surface. One important factor
1634 which influences binding capacity is the type of treatment that a surface has received. When
1635 working with a surface treated with a nonpolar wax, such as that used on floor tile, a nonpolar
1636 compound will be adsorbed to the surface, which further limits recovery. In contrast, recovery
1637 from absorbent surfaces, such as lab bench paper or untreated wood, may give poor recoveries
1638 due to the porous nature of the surface.

1639 **10.6.2 Sample Handling**

1640 Filter paper or other materials used for wipe tests in the field should be placed in separate
1641 containers that prevent cross contamination during transport and allow for labeling of each
1642 sample. Plastic bags, paper or glassine envelopes, and disposable plastic petri dishes are
1643 containers typically used to store and transport wipe samples. Field workers can use plastic or
1644 rubber gloves and forceps when applying the wipe material to a surface and during handling as
1645 each wipe is placed into a container. Protection of the sample wipe surface is the main concern
1646 when a wipe must be placed in a container for transport. If a scintillation vial or planchet will be
1647 used in the lab, then a field worker may put wipes directly into them. Planchets containing loose
1648 or self-sticking wipes can also be put into self-sealing plastic bags to separate and protect the

1649 integrity of the sample's surface. Excessive dust and dirt can cause self adsorption or quenching,
1650 and therefore should be minimized.

1651 To maintain constant geometry in an automatic proportional counter, it is important that the wipe
1652 remain flat during counting. Additionally, material that will curl can jam the automatic counter
1653 and cause cross contamination or even destroy the instrument window. When it is necessary to do
1654 destructive analysis on the wipe, it is critical that the wipe can easily be destroyed during the
1655 sample preparation step, and that the residue not cause interference problems.

1656 When wipes are put directly into liquid scintillation cocktail, it is important that the wipe not add
1657 color or react with the cocktail. For maximum counting efficiency, as well as reproducibility, the
1658 wipe should either dissolve or become translucent in the cocktail.

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