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Standard Guide for Rapid Screening of Vegetation for Radioactive Strontium Aerial Deposition[1]

This standard is issued under the fixed designation D7362; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This guide provides a rapid procedure by which vegetation samples may be screened for surface contamination of radioactive strontium (^{89}Sr and ^{90}Sr, collectively referred to as radiostrontium) following an airborne radioactive dispersal event. It provides a conservative estimate of radiostrontium deposition that can be used by decision makers for immediate actions prior to obtaining definitive results from a fixed laboratory asset.

1.2 Insoluble forms of radiostrontium, such as the strontium (^{90}Sr) titanate ($SrTiO_3$) used in radio-isotope thermal-electric generators (RTGs), will not be measured by this method.

1.3 Non-SI units are used in the calculations of this guide for ease of use during the emergency phase of an event. The instrumentation used typically provides count rates in counts per minute (cpm) rather than per second (s^{-1}, the SI unit), thus activity is expressed in dpm (decays per minute) rather than Bq. Additionally, US EPA protective guidelines for surface contamination are expressed in dpm/100 cm^2.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1129 Terminology Relating to Water

D1193 Specification for Reagent Water

D3648 Practices for the Measurement of Radioactivity

2.2 *Other Documents:*

EPA Protective Action Guidelines [3]

3. Terminology

3.1 *Definitions*—See Terminology D1129 for terms related to water.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *ROI, n*—region of interest; the span of channels, or region, in the spectrum in which the counts due to a specific radioisotope appear on a functioning, calibrated liquid scintillation spectrometry system.

3.3 *Acronyms:*

3.3.1 *RLS, n*—rapid liquid sampler

3.3.2 *SPE, n*—solid phase extraction

4. Summary of Guide

4.1 Vegetation is collected from an area equivalent to 100 cm^2. The leafy material is shaken with pH = 2 water to solubilize radiostrontium deposited on the vegetation. The radiostrontium is then extracted onto a solid phase extraction (SPE) disk for counting and quantification.

4.2 Testing has shown that chemical recoveries for ^{90}Sr under these extraction conditions average 30–50 %, with similar recoveries expected for ^{89}Sr.

4.3 A counting efficiency of 80–85 % can be achieved using liquid scintillation spectrometry.

4.4 Quantification may also be accomplished using a simple gas-filled count rate meter (a "pancake probe"); however the presence of other beta-emitting radionuclides can not be discerned when using such a non-discriminatory detector.

5. Significance and Use

5.1 Strontium-90 is a major component of nuclear waste and is also a potential radioisotope for use as a weapon of mass destruction in a radiological dispersal device. It is a beta-emitting radioisotope with moderate half-life (~30 years).

[1] This guide is under the jurisdiction of ASTM Committee D19 on Water and is the direct responsibility of Subcommittee D19.04 on Methods of Radiochemical Analysis.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from United States Environmental Protection Agency (EPA), Ariel Rios Bldg., 1200 Pennsylvania Ave., NW, Washington, DC 20460, http://www.epa.gov.

Strontium-89 is also a beta emitting radionuclide, but with a half-life of only ~50 days it is not usually present in significant quantities. If ingested the radiostrontium may deposit in the bone of an individual and thus can contribute a significant radiological dose to an affected person.

5.2 Following an explosion in which radioactive material was present, the potential exists for the material to become airborne. It will quickly attach to atmospheric particles and be deposited on surfaces as the plume passes. This guide provides a rapid procedure by which vegetation can be screened to determine if radiostrontium is present and to provide a conservative estimate of its deposition on vegetation.

5.3 This guide is intended to be used in a field portable lab, or if needed, can be performed completely in the field; therefore no hazardous chemicals are required to complete the analysis. However, an option for the use of acid in certain steps is documented in this guide.

5.4 This guide is not intended to be used for screening food products or animal feed following an accident or incident.

6. Interferences

6.1 Liquid scintillation (LS) counting is the preferred method of counting. Because this is a screening method, chemical decontamination from other beta-emitting isotopes is not as rigorous as found in traditional laboratory methods. Careful evaluation of the liquid scintillation spectrum may provide indications of the presence of contaminants.

6.2 Lead and radium are known to also be retained by the SPE disk under these extraction conditions and do occur naturally as part of fallout deposition, although at low concentrations compared to the radiostrontium surface contamination of concern. Careful evaluation of the liquid scintillation spectrum may provide indications of the presence of alpha peaks (Ra isotopes) or other beta continuums (Pb or other isotopes).

6.3 Yttrium-90, the daughter of ^{90}Sr and also a beta emitter, will be partially retained by the SPE disk using this method. If permissible, and with appropriate personal protective equipment and engineering controls, the SPE disk can be washed with 15–20 mL of 2-3 M nitric acid. Note the time the wash finished going through the SPE disk as the start time for ^{90}Y ingrowth.

6.4 Particles containing ^{90}Sr (such as from an RTG explosion), as opposed to particles with radiostrontium adsorbed on the surface, may not be sufficiently attacked by the weak acid solution and so will not be detected by this procedure. However, particles containing ^{90}Sr would only be expected to be encountered very close to the site of the initial explosion.

7. Instrumentation

NOTE 1—See Practices D3648 for a description of these detector systems.

7.1 *Liquid Scintillation Spectrometer*—Commercial systems are available that are reasonably portable, but may require manual sample changing. The system should have a stable background in the counting region of interest and reproducible detection efficiency for ^{90}Sr on the SPE disk. The spectrometer should allow the option of looking at the entire counting spectrum so that evaluation of other interferences may be completed. Automatic discrimination of alpha and beta particles is desirable but not required.

7.2 *Gas Filled Count Rate Meter*—A non-discriminating beta/gamma radiation detector, often called a "pancake probe," consisting of a gas-filled detector coupled to an electronics package that provides count rate information.

8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[4]

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean Type I water as defined in Specification D1193.

8.3 *Solid Phase Extraction (SPE) Disk*—47 mm in diameter, impregnated with strontium selective resin,[5] supplied as either loose SPE disks or in the Rapid Liquid Sampler (RLS) housing.

8.4 *pH = 2 Water*—Water acidified to a pH of 2 with nitric acid. Concentrated nitric acid may be added dropwise, mixing and monitoring the pH of the water until it begins to approach 2–4, then carefully add a more dilute nitric solution (1–2 M) until a pH of 2 is obtained. If the pH is overshot, add a dilute Na_2CO_3, $NaHCO_3$ or NaOH solution to a final pH of 2.

8.5 *Nitric Acid*—(HNO_3) concentrated and diluted solution.

8.6 *Liquid Scintillation (LS) Cocktail*—Any commercial cocktail that provides acceptable counting efficiency and backgrounds in the ROI for $^{89+90}$Sr, many are deemed environmentally friendly thus do not require disposal as a mixed waste.

8.7 *Strontium-90 Calibration Solution*—A solution whose activity is traceable to a national standards body, such as NIST or NPL, in dilute nitric acid. Alternatively, ^{89}Sr may be used but the known activity must be decay corrected to the time of use.

9. Apparatus

9.1 Wide mouth plastic bottle, 1–2 L preferred or 1–2 qt resealable plastic bags (that is, bags with a zipper-locking seal).

9.2 Device for measuring or delivering approximately 50-mL aliquots of acidified water.

[4] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals,* BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary,* U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

[5] The sole source of supply of the apparatus known to the committee at this time is trademark Strontium Empore solid phase extraction disk or equivalent, 3M Company, St. Paul, MN, http://www.3m.com. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

9.3 60-mL syringe, disposable, plastic.

9.4 47-mm filter holder with Leur-Lock fitting (not required if the SPE disks are packaged in the RLS housing).

9.5 Tin snips or wire cutters used to open the RLS housing of the SPE disk (not required if using disks in the 47-mm filter holder).

9.6 Liquid scintillation counting vials (glass or plastic), 20-mL, or as appropriate for the detector to be used.

9.7 Sample holder for the SPE disks for counting by gas-filled detector: small resealable plastic bags (that is, bags with a zipper-locking seal) or pouches made from thin aluminum foil to prevent the spread of contamination when counting the SPE disks.

9.8 pH-indicating test strips (to include a range of 1–8) or calibrated pH meter.

10. Calibration and Standardization

10.1 *Detector Efficiency:*

10.1.1 The liquid scintillation (LS) spectrometry system should be calibrated for background and detection efficiency. Typical ROIs for the determination of ^{90}Sr are 2–500 keV and 500–2000 keV. The ^{90}Sr will be in the lower window; indications of ^{90}Y will be seen as counts in the higher energy window.

10.1.1.1 The background count rate is determined by placing a blank Strontium SPE disk in a LS counting vial with cocktail and counted using the appropriate ROIs.

10.1.1.2 To determine the counting efficiency for ^{90}Sr on the SPE disk a known amount of ^{90}Sr, in equilibrium with ^{90}Y, is added to ~50 mL of DI water and the pH adjusted to 2 as described in 8.4. The solution is poured through a SPE disk (for calibration the syringe method may be used or a vacuum filtration apparatus may be used). The disk is placed in a LS counting vial with cocktail and counted using the preset ROIs.

NOTE 2—The waste solution can be saved, reduced in volume and counted by LSC to confirm quantitative recovery of the ^{90}Sr on the calibration disk.

10.1.1.3 The ratio of the net counts per minute (cpm) in the ^{90}Sr ROI to the known activity added (in decays per minute, dpm) is the fractional detection efficiency (ε):

$$\varepsilon = \frac{R_s - R_b}{A_{s\epsilon}} \tag{1}$$

where:

R_s = gross count rate in ^{90}Sr ROI (in cpm),
R_b = background count rate in ^{90}Sr ROI (in cpm), and
$A_{s\varepsilon}$ = spike activity added (in dpm).

10.1.1.4 Prepare at least three calibration disks and use the average in all subsequent calculations.

10.1.2 If a gas-filled detector is to be used for the field screening analyses, the counting efficiency may be determined by counting the disk prepared above with the appropriate detector. The ratio of the net count rate (in cpm) to the known spike addition (in dpm) is an indication of counting efficiency (see Eq 1).[6]

10.2 *Chemical Recovery:*

10.2.1 Because the samples are analyzed with no yield monitor, an average chemical recovery is determined in the lab and applied to the rapid field analyses.

10.2.2 Leafy vegetation should be collected from an area known to contain no radioactive contamination. It should be a common variety so that it will be applicable to emergency response actions.

10.2.3 An amount of vegetation equivalent to 100 cm^2 collection area is placed in a plastic bottle or bag.

10.2.4 A known amount of ^{90}Sr spike solution is dispensed directly onto the vegetation in the bottle and then the extraction procedure, below, is followed.

10.2.5 The samples are counted on either the LS system or using the gas-filled detector. The ratio of the measured net count rate (in cpm), corrected for counting efficiency, to the known spike addition (in dpm) is an indication of the fractional chemical recovery (Y):

$$Y = \frac{R_s - R_b}{\varepsilon \times A_{sY}} \tag{2}$$

where:

R_s = gross count rate in ^{90}Sr ROI (in cpm),
R_b = background count rate in ^{90}Sr ROI (in cpm),
ε = counting efficiency (calculated in Eq 1), and
A_{sY} = spike activity added (in dpm).

10.2.6 Several spiked samples should be completed in the laboratory to obtain an average chemical yield for field analyses.

11. Procedure

11.1 Collect vegetation from an area of concern, preferably leafy material out in the open. Vegetation should be collected from an area roughly equivalent to 100 cm^2. Use gloves; try not to dislodge any dust off the leaves while collecting as much of the vegetation as possible from the sampling area. Stems and other material close to the ground that may not have deposition do not need to be collected. A pair of grass clippers may be useful to aid in collecting the sample.

11.2 Place the vegetation in a wide-mouth 1–2 L plastic bottle. The bottle should be no more than about 10–25 % full. Alternatively, the sample could be placed in a 1 or 2-qt resealable plastic bag (that is, a bag with zipper-locking closure).

11.3 Add approximately 50 mL of pH = 2 water.

11.4 Cap and shake the bottle. Make sure the acidic solution comes in contact with as much of the leafy material as possible. Shake for at least 3 min.

[6] Beals, D.M., Hofstetter, K.J., Johnson, V.G., Patton, G.W., Seely, D.C., "Development of field portable sampling and analysis systems," *Journal of Radioanalytical and Nuclear Chemistry*, Vol 248, No. 2, 2001, pp. 315–319.

11.5 Place a 60-mL syringe on a SPE disk holder (either the RLS format or 47-mm filter holder with Luer-Lock fittings containing the disk). Pull the plunger out of the syringe.

11.6 Decant the water from the sample bottle into the syringe. Replace the plunger and push the solution through the SPE disk. Collect the waste in a container designated for waste.

11.7 Remove the syringe from the SPE disk holder and pull the plunger out then reattach the syringe to the SPE disk holder without the plunger.

11.8 Add an additional ~50 mL of pH = 2 water to the original sample bottle, cap and shake for at least 3 min longer.

11.9 Decant the solution from the sample bottle into the same syringe as used for the first wash. Replace the plunger and push the solution through the SPE disk. Collect the waste as before.

11.10 Remove the syringe from the SPE disk holder. Pull the plunger part-way out (to the 30–40 mL mark), reattach the syringe to the SPE disk holder and push air through the disk holder to remove excess water.

11.11 Remove the syringe from the disk holder then carefully remove the SPE disk from the holder. When opening the commercial RLS housing a pair of tin snips or wire cutters have been found suitable.

11.12 Place the SPE disk into a 20–mL LS vial, add 20 mL of cocktail and shake well, or place the disk in a holder (such as a small bag or thin foil pouch[6]) for counting using a gas-filled detector.

11.13 Count the disk.

11.13.1 To provide information as quickly as possible, the sample may initially be counted for about 0.5–1 min using the gas-filled detector. If the sample count rate is above the instrument background count rate by 3 times or more, consider reporting that the sample has elevated activity.

11.13.2 Next, remove the sample from the holder and place into a LS vial for a more definitive count. Count the sample for several minutes and evaluate the spectrum to ascertain if the principal component is radiostrontium or some other beta-emitting radioisotope.

12. Calculations

12.1 *Calculation of Activity*—If the area sampled can be estimated (recommended 100 cm^2 in this guide) a conservative value can be calculated for the deposition of ^{90}Sr by:

$$A_A = \frac{R_s - R_b}{Y_{avg} \times \varepsilon \times A} \quad (3)$$

where:

A_A = areic activity of ^{90}Sr (dpm/cm^2),
Y_{avg} = average chemical recovery (determined in the lab), and
A = area sampled (cm^2).

NOTE 3—Because this is a screening method no rigorous estimation of uncertainty is useful. Counting error may be estimated as a percentage of the final result based on the fluctuation in the gas-filled count rate meter response, or as the square root of counts over time for the LS detector.

12.2 *Minimum Detectable Concentration*—An estimation of the MDC for this method can be calculated by:

$$MDC = \frac{2.71 + 4.65\sqrt{C_b}}{\varepsilon \times Y \times A \times t_b} \quad (4)$$

where:

C_b = counts in background in time t_b, and
t_b = count time in min.

Values that have been obtained using this guide are presented in Table 1.

13. Precision and Bias

13.1 This procedure is offered as a guide with options for counting the sample therefore no precision and bias study has been completed. The lack of a suitable standard precludes a bias study. Users of this guide may submit their findings to the ASTM committee responsible for this guide. If sufficient information is received the committee may seek to revise this guide as a standard method.

14. Keywords

14.1 emergency response; Liquid Scintillation Counting; Solid Phase Extraction; strontium-89; strontium-90; vegetation

TABLE 1 Values Obtainable Using this Guide

Detector	Background cpm	Count time minute	Counts in backgroud	Efficiency %	Yield %	MDC dpm/ sample
beta-gamma probe	50	0.25	12.5	50	30	511
portable LSC	20	10	200	80	30	29