Designation: C999 – 17

Standard Practice for Soil Sample Preparation for the Determination of Radionuclides[1]

This standard is issued under the fixed designation C999; 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 practice covers the preparation of surface soil samples collected for analysis of radionuclide constituents, particularly uranium and plutonium. This practice describes one acceptable approach to the preparation of soil samples for radiochemical analysis.

1.2 The values stated in SI units are to be regarded as standard. The values given in parentheses are for information only.

1.3 *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.* A specific hazard statement is given in 7.3.

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C859 Terminology Relating to Nuclear Materials

C998 Practice for Sampling Surface Soil for Radionuclides

C1402 Guide for High-Resolution Gamma-Ray Spectrometry of Soil Samples

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

3. Terminology

3.1 Except as otherwise defined herein, definitions of terms are as given in Terminology C859.

4. Summary of Practice

4.1 Guidance is provided for the preparation of a homogeneous soil sample from ten composited core samples (aggregate weight of 4 to 5 kg) collected as to be representative of the area.

5. Significance and Use

5.1 Soil samples prepared for radionuclide analyses by this practice can be used to characterize radionuclide constituents. This practice is intended to produce a homogeneous sample from which smaller aliquots may be drawn for radionuclide characterization.

5.2 Many soil characterization plans for radionuclide constituents utilize gamma-ray spectrometry measurements of soil to quantify a number of possible gamma emitting analytes. A widely used practice for these measurements is to fill a calibrated sample container, such as a Marinelli beaker (~600-mL volume), with a homogenized soil sample for counting such as what may be done using Guide C1402. By preparing the entire soil core collection, sufficient homogeneous sample is available for such gamma-ray spectrometry and other radiochemical measurements.

6. Apparatus

6.1 *Scale,* capacity of 10 kg.

6.2 *Drying Oven,* able to maintain ±2°C.

6.3 *Pans,* disposable aluminum.

6.4 *Jar Mill,* capacity for 7.57-L (2-gal) cans.

6.5 *Steel Cans and Lids,* 7.57-L (2-gal).

6.6 *Ceramic Rods,* 21 by 21-mm (13⁄16 by 13⁄16-in.) or steel grinding balls, 25.4-mm (1-in.) diameter.

6.7 *Sieve,* U.S. Series No. 35 (500-µm or 32 mesh).

6.8 *Plastic Bottles,* 7.57-L (2-gal).

[1] This practice is under the jurisdiction of ASTM Committee C26 on Nuclear Fuel Cycleand is the direct responsibility of Subcommittee C26.05 on Methods of Test.

Current edition approved June 1, 2017. Published July 2017. Originally approved in 1983. Last previous edition approved in 2010 as C999 – 05 (2010)ε1. DOI: 10.1520/C0999-17.

[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.

7. Procedure

7.1 Label a cleaned 7.57-L (2–gal) steel can and lid with a unique laboratory code number.

7.2 Weigh the labeled steel can and lid. Record the weight.

7.3 Transfer the ten soil cores (including vegetation) from the field collection containers, such as may have been collected using Practice C998, into the labeled, preweighed steel can. Do not pack the can full. Place the steel lid loosely on the can. (**Warning**—Wear gloves throughout the preparation procedure to minimize the possibility of fungus infection.)

7.4 Weigh the sample cores, steel can, and lid to ±50 g. Record the weight.

7.5 Remove the lid and place the sample in a 110°C drying oven for 24 h or longer, depending on the depth of soil in the can, until the sample has reached constant weight.

7.6 Remove the sample from the oven, cap the can with its lid, and cool to room temperature.

7.7 Weigh the dried sample cores, steel can, and lid to ±50 g. Record the weight.

7.8 Remove the can lid and add 10 to 12 ceramic rods (21 by 21-mm) or steel balls (25.4–mm diameter) to the can.

7.9 Replace the lid and tightly seal the sample can.

7.10 Place the sample can on a jar mill for at least 4 h, or overnight if possible, at 30 r/min.

7.11 Remove the sample can from the mill and place in a hood.

7.12 Allow the sample to settle for a few minutes.

7.13 Label a 7.57-L (2-gal) plastic jar and cap with the laboratory code number of the sample.

7.14 Remove the lid from the sample can and transfer a portion of the sample to a U.S. Series No. 35 (500-µm or 32 mesh) sieve.

7.15 Sieve the sample and transfer the sieved fraction to the prelabeled plastic jar.

7.16 Repeat the sieving and transfer steps until the entire sample has been processed.

7.17 Remove the ceramic rods or steel balls from the unsieved material.

7.18 Place the unsieved material in the can and replace the lid.

7.19 Weigh, record the weight, and discard the unsieved material and can. (**Caution**—The unsieved material should consist of rocks, stones, sandy matter, and any remaining vegetation. If soil clumps remain, additional milling is required.) (**Caution**—The ceramic or steel grinding media and the sieve must be cleaned thoroughly prior to reuse to eliminate the possibility of cross-contamination of samples.)

7.20 Remove a suitable aliquot of the sample from the jar for radiochemical analysis using for example Guide C1402.

7.21 Cap the sample jar tightly. Wash and dry the outside of the container prior to storage.

8. Calculation

8.1 *Wet Weight of the Composited Soil Cores*—The wet weight (W) of the composited soil cores is the weight measured prior to oven-drying the cores as follows:

$$W = T - C \tag{1}$$

where:

W = wet weight of the composited soil cores, g,
T = weight of the soil cores, steel can, and lid, g (from 7.4), and
C = weight of the empty steel can and lid, g (from 7.2).

8.2 *Dry Weight of the Composited Soil Cores*—The dry-weight (D) of the composited soil cores is the weight measured after drying the cores at 110°C as follows:

$$D = N - C \tag{2}$$

where:

D = dry (110°C) weight of the soil cores, g,
N = weight of the dried (110°C) soil cores, steel can, and lid, g (from 7.7), and
C = weight of the empty steel can and lid, g (from 7.2).

8.3 *Bulk Density of the Soil Cores*—The bulk density (B) of the soil cores may be estimated from the wet weight of the cores (W) and the number of cores collected for compositing, times the volume of the sampling corer used in the field collection.

$$B = (W)/(F \times V) \tag{3}$$

where:

B = bulk density of the composited soil cores, g/cm^3,
W = weight of the composited soil cores, g, (from 8.1),
F = number of soil cores collected and composited (10 cores in accordance with Practice C998), and
V = volume of sampling corer used for the field collection, cm^3.

8.4 *Weight of Unsieved Material*—The weight of the unsieved material, consisting primarily of rocks and stones, is obtained for documentation purposes.

9. Keywords

9.1 environmental; preparation; radionuclides; soil

APPENDIX

(Nonmandatory Information)

X1. RATIONALE

X1.1 A soil sampling and analysis program provides a direct means of determining the concentration and distribution pattern of radionuclides in the environs of nuclear facilities.[3]

X1.2 This practice was developed to minimize sample handling and economic costs while providing a final sample homogeneity adequate for the intended radiochemical analyses. For these reasons, the soil cores collected in the field are treated as a single sample without preliminary subdivision into arbitrary fractions, such as +2-mm or −2-mm sizes. Vegetation is not separated from the cores because it contributes little to the volume or bulk density of the sample. Rocks and stones allowed to remain in the sample during the milling operation act as additional grinding media. After the milling operation, the rocks and stones may be discarded because these materials would not contain radionuclides originating from a nuclear facility release.

X1.3 The milling of the soil to No. 35 (500-µm or 32 mesh, see Table X1.1) sieve size is based on consideration of the particle size of plutonium present in soil at three sites of releases. Tamura[4] developed empirical information which shows that essentially 100 % of the plutonium is present in the No. 35 sieve fraction. Also see Specification E11.

TABLE X1.1 Various Sieve Size Designations

U.S. Series Designation		Tyler Screen Scale Equivalent	Sieve Opening, in. (approximate equivalent)
Alternative	Standard		
No. 4	4.75 mm	4 mesh	0.187
No. 6	3.35 mm	6 mesh	0.132
No. 8	2.36 mm	8 mesh	0.0937
No. 10	2.00 mm	9 mesh	0.0787
No. 12	1.70 mm	10 mesh	0.0661
No. 14	1.40 mm	12 mesh	0.0555
No. 16	1.18 mm	14 mesh	0.0469
No. 18	1.00 mm	16 mesh	0.0394
No. 20	850 µm	20 mesh	0.0331
No. 30	600 µm	28 mesh	0.0234
No. 35	500 µm	32 mesh	0.0197
No. 40	425 µm	35 mesh	0.0165
No. 45	355 µm	42 mesh	0.0139
No. 50	300 µm	48 mesh	0.0117
No. 60	250 µm	60 mesh	0.0098
No. 70	212 µm	65 mesh	0.0083
No. 80	180 µm	80 mesh	0.0070
No. 100	150 µm	100 mesh	0.0059
No. 120	125 µm	115 mesh	0.0049
No. 140	106 µm	150 mesh	0.0041
No. 170	90 µm	170 mesh	0.0035
No. 200	75 µm	200 mesh	0.0029
No. 230	63 µm	250 mesh	0.0025
No. 270	53 µm	270 mesh	0.0021
No. 325	45 µm	325 mesh	0.0017

[3] "Measurements of Radionuclides in the Environment: Sampling and Analysis of Plutonium in Soil," Atomic Energy Commission Regulatory Guide 4.5, May 1974.

[4] Tamura, T., "Physical and Chemical Characteristics of Plutonium in Existing Contaminated Soils and Sediments," Proceedings of the Symposium on Transuranium Nuclides in the Environment, IAEA Pub ST1/PUB/410, Vienna, 1976.