Standard Practice for Soil Sample Preparation for the Determination of Radionuclides¹

This standard is issued under the fixed designation C 999; 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 chemical analysis of radionuclides, particularly uranium and plutonium. This practice describes one acceptable approach to the preparation of soil samples for radiochemical analysis.

1.2 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 Note 1.

2. Referenced Documents

2.1 ASTM Standards:

C 998 Practice for Sampling Surface Soil for Radionuclides²

3. Summary of Practice

3.1 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.

4. Significance and Use

4.1 Soil samples prepared for radionuclide analyses by this practice are used to monitor fallout distribution from nuclear facilities. This practice is intended to produce a homogeneous sample from which a relatively small aliquot (10 g) may be drawn for radiochemical analyses.

4.2 Most nuclear facilities fulfill major requirements of their monitoring programs by gamma-ray spectrometry measurements of soil. A widely used practice for these measurements is to fill a calibrated sample container, such as a Marinelli beaker (\sim 600-mL volume), with a homogenized soil sample. By preparing the entire soil core collection, sufficient homogeneous sample is available for radiochemical and gamma-ray spectrometry measurements.

5. Apparatus

- 5.1 Scale, capacity of 10 kg.
- 5.2 Drying Oven, able to maintain $\pm 2^{\circ}$ C.
- 5.3 Pans, disposable aluminum.
- 5.4 Jar Mill, capacity for 7.57-L (2-gal) cans.
- 5.5 Steel Cans and Lids, 7.57-L (2-gal).

5.6 *Ceramic Rods*, 21 mm by 21 mm (13/16 in. by 13/16 in.)

- or steel grinding balls, 25.4-mm (1-in.) diameter.
 - 5.7 Sieve, U.S. Series No. 35 (500-µm or 32 mesh).
 - 5.8 Plastic Bottles, 7.57-L.

6. Procedure

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

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

6.3 Transfer the ten soil cores (including vegetation) from the field collection containers into the labeled, preweighed steel can. Do not pack the can full. Place the steel lid loosely on the can.

NOTE 1—**Precaution:** Wear gloves throughout the preparation procedure to minimize the possibility of fungus infection.

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

6.5 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.

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

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

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

6.9 Replace the lid and tightly seal the sample can.

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

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

6.12 Allow the sample to settle for a few minutes.

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

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

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6.15 Transfer the sieved fraction to the prelabeled plastic jar.

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

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

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

6.19 Weigh, record the weight, and discard the unsieved material and can.

NOTE 2—Caution: The unsieved material should consist of rocks, stones, and sandy matter. If soil clumps remain, additional milling is required.

NOTE 3—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.

6.20 Remove a suitable aliquot of the sample from the jar for radiochemical analysis.

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

7. Calculation

7.1 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:

$$W = T - C$$

where:

W = wet weight of the composited soil cores, g,

- T = weight of the soil cores, steel can, and lid, g (from 6.4), and
- C = weight of the empty steel can and lid, g (from 6.2).

7.2 Dry Weight of the Composited Soil Cores—The dryweight (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:

 $D = dry (110^{\circ}C)$ weight of the soil cores, g,

N = weight of the dried (110°C) soil cores, steel can, and lid, g (from 6.7), and

C = weight of the empty steel can and lid, g (from 6.2).

7.3 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.

$$B = (W)/(F \times V)$$

(3)

where:

B = bulk density of the composited soil cores, g/cm³,

W = weight of the composited soil cores, g,

F = number of soil cores collected and composited (10 cores in accordance with Practice C 998), and

V = volume of sampling corer used for the field collection, cm³.

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

8. Keywords

8.1 environmental; preparation; radionuclides; soil

APPENDIX

(1)

(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- μ m 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. Tamura⁴ developed empirical information which shows that essentially 100 % of the plutonium is present in the No. 35 sieve fraction.

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.

TABLE	X1.1	Various	Sieve	Size	Designations
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U.S.	Series Designation	on Ty Scr	ler Sieve	Sieve Opening,	
Alternativ	ve Stan	dard Sc Equiv	ale eq valent eq	uivalent)	
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. 1	0 2.00	mm 9	mesh (0.0787	
No. 1	2 1.70	mm 10	mesh (0.0661	
No. 1	4 1.40	mm 12	mesh ().0555	
No. 1	6 1.18	mm 14	mesh (0.0469	
No. 1	8 1.00	mm 16	mesh (0.0394	
No. 2	0 850	μm 20	mesh (0.0331	
No. 3	0 600	μm 28	mesh (0.0234	
No 3	5 500	um 32	mesh (0197	
No. 4	0 425	um 35	mesh (0 0 1 6 5	
No 4	5 355	um 42	mesh (0 0139	
No. 5	0 300	um 48	mesh (0.0117	
No. 6	0 250	µm 60	mesh (0.0098	
No 7	0 212	um 65 i	mesh (0.0083	
No. 8	0 212	um 80.	mesh (0070	
No. 10	0 100	um 100 l	mesh (0.0070	
No. 12	0 125	um 115 i	mesh (0.0000	
No. 14	.0 106	um 150	mesh (0.0040	
110. 11		pin 100 i			
No. 17	0 90	μm 170	mesh (0.0035	
No. 20	0 75	μm 200	mesh (0.0029	
No. 23	63	μm 250	mesh (0.0025	
No. 27	0 53	μm 270	mesh (0.0021	
No. 32	5 45	μm 325	mesh (0.0017	

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