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Final

PLUTONIUM IN SOILS TREATABILITY STUDIES
Work Plan: TRUclean PROCESS

ROCKY FLATS PLANT
OPERABLE UNIT 2

U. S. DEPARTMENT OF ENERGY
ROCKY FLATS PLANT
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ENVIRONMENTAL RESTORATION MANAGEMENT

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Plutonium in Soils Treatability Studies Work Plan -TRU Clean Process

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7.0 TRUclean PROCESS

The TRUclean Process technology was selected during the development of the TSP for bench-scale treatability testing as a result of a screening process designed to identify candidate technologies for use in corrective/remedial actions at the RFP (DOE, 1991). **The following information on the TRUclean Process is proprietary to LES&TC.**

7.1 Type of Treatability Study

The treatability study to be conducted with the TRUclean Process is considered "bench-scale". The equipment is located at LES&TC treatability study laboratory. Operators at LES&TC will attempt to optimize the performance of the TRUclean Process with untreated RFP soil by varying process parameters such as bed depth of the gravimetric separator (GS), solids/liquid ratio in the attrition scrubber, and pH of wash solution. A detailed description of the TRUclean Process and its associated test procedures are included in following sections of this work plan.

7.2 Technology Description

The key component of the TRUclean Process to be tested as part of this treatability study is a Gravimetric Separator (GS). A GS separates materials with different specific gravities by flowing a slurried material over a screen and screen bed subjected to a vertical hydraulic pulse. The pulse is the result of a sudden upflow of water through the screen and screen bed with sufficient velocity to bring all particles momentarily into suspension so that the screen bed becomes fluidized. At the completion of the pulse, the water drains back through the screen and screen bed and the pulse cycle is repeated. The pulsing cycle allows heavy particles to settle through the screen bed and lighter particles to pass over the top of the screen bed. Heavy particles that are small enough to pass through the screen are discharged continuously. Coarse, heavy particles not passing through the screen are withdrawn intermittently. The lighter material flows over the top of the screen bed and out of the end of the GS.

There are three product streams from a GS: low density tailing which is skimmed over the weir; oversize dense particles which collect on the screen; and dense fines or "hutch product" which pass through the screen. The oversize dense particle stream is not a continuously generated material; it is removed on a periodic basis. Separation is accomplished by the different settling rates of the particles in a verticle pulse of water. Figure 7.2-1 provides a simple diagram for the components and streams of a typical GS. Figure 7.2-2 provides an actual size, visual representation of various particle sizes from coarse to fine.

The ability of a GS to achieve fractionation by particle density and particle size is what provides an isolation of plutonium contamination. Since plutonium (PuO_2) has a high specific gravity (Sp. Gr. = 11.5) and is

associated with fines (DOE, 1982), it is expected that the plutonium contamination will be concentrated primarily in the hutch product stream.

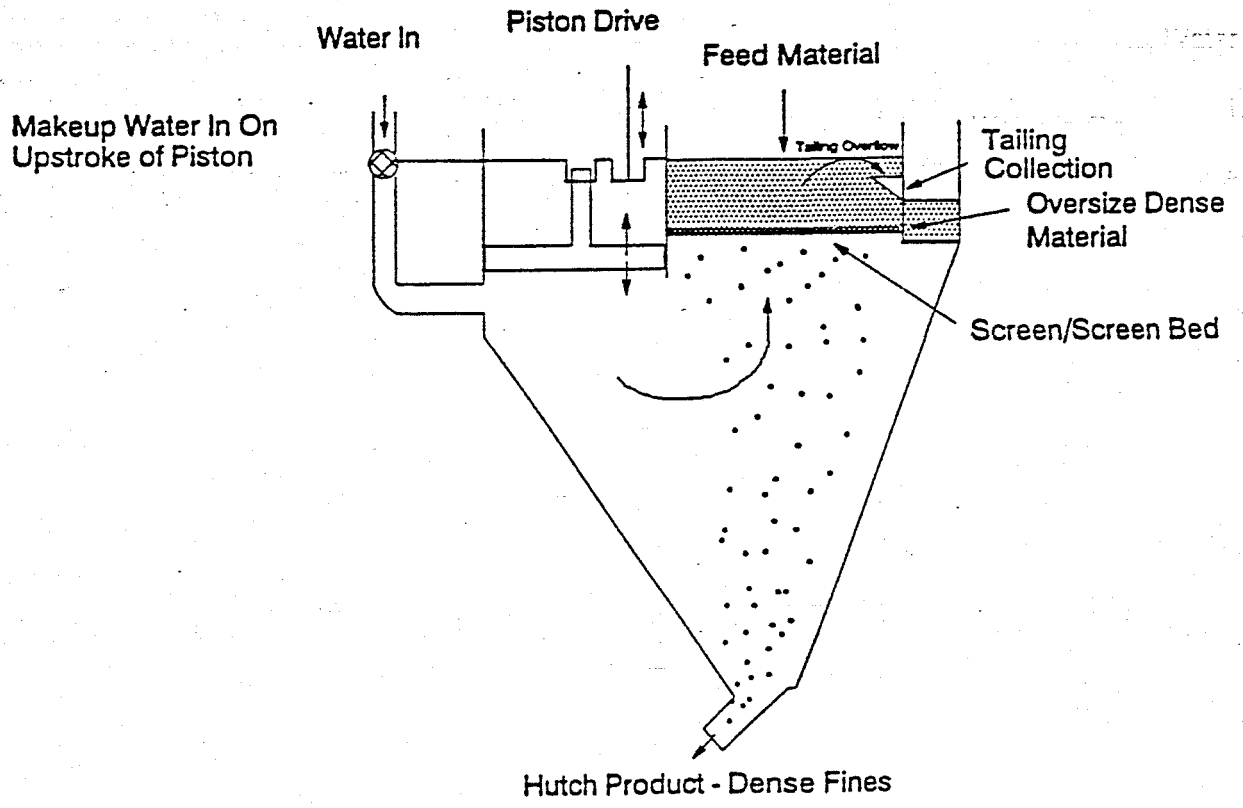
To enhance the performance of the TRUclean Process GS, several feed preparation steps have been added. These steps include an initial wet screening of the soil by a trommel screen, attrition scrubbing to promote separation of plutonium particulates from the soil aggregate, and particle size fractionation by hydrocyclones. The hydrocyclones are optional and will be used if improvement is needed in effecting particle size cuts. Also, unit operations have been added to "polish" the tailing stream of the GS in an effort to further isolate the plutonium contamination. These unit operations include: particle size fractionation by spiral classifier and gravimetric separation by centrifugal concentrator.

Figure 7.2-3 presents a general flow diagram showing the typical arrangement of unit operations making up the TRUclean Process. A detailed description of the operational procedures and equipment for the test process (with modifications to the general TRUclean Process flow) as applied to the RFP plutonium-contaminated soil is included in Section 7.3.4 of this work plan.

7.3 Experimental Procedures and Equipment

LES&TC will conduct a treatability test on RFP plutonium-contaminated soils. The goal of the experimental procedures is to select the bench-scale process equipment (Phase I) and place that equipment in the most effective sequence (Phase II) to separate clean (less than 0.9 pCi/g Plutonium 239 plus Plutonium 240) soils from plutonium-contaminated soils.

There are at least eight pieces or types of process equipment that will be tested. There are at least five additional types of support equipment that



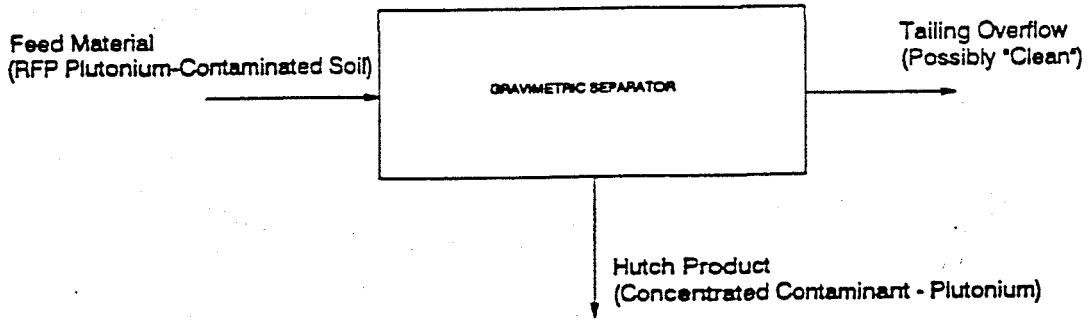


FIGURE 7.2-1

GENERAL CONFIGURATION AND FLOW

FOR A GRAVIMETRIC SEPARATOR

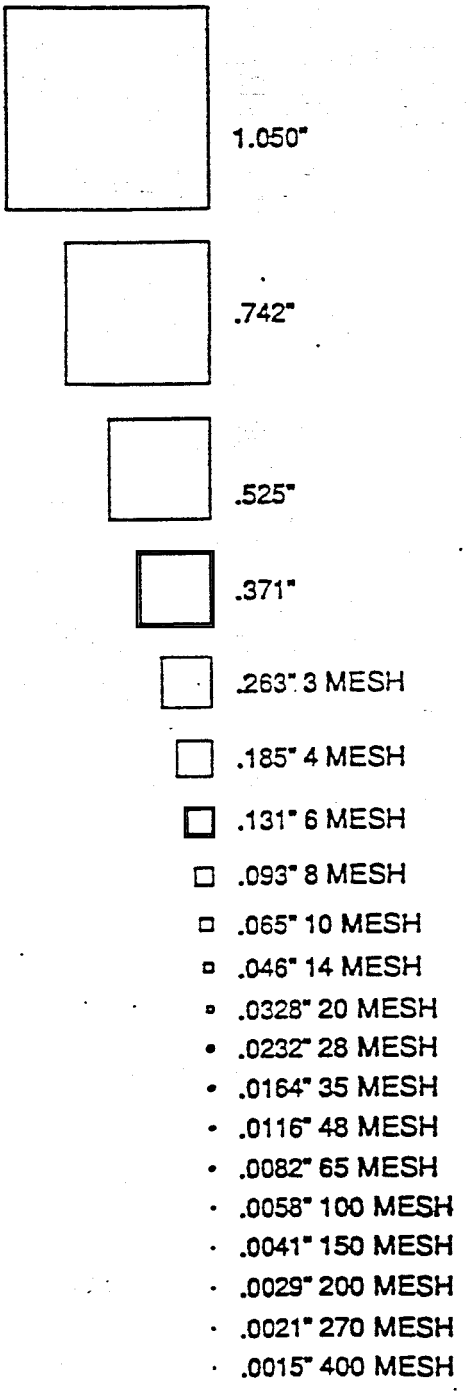


FIGURE 7.2-2

PARTICLE SIZE REPRESENTATION

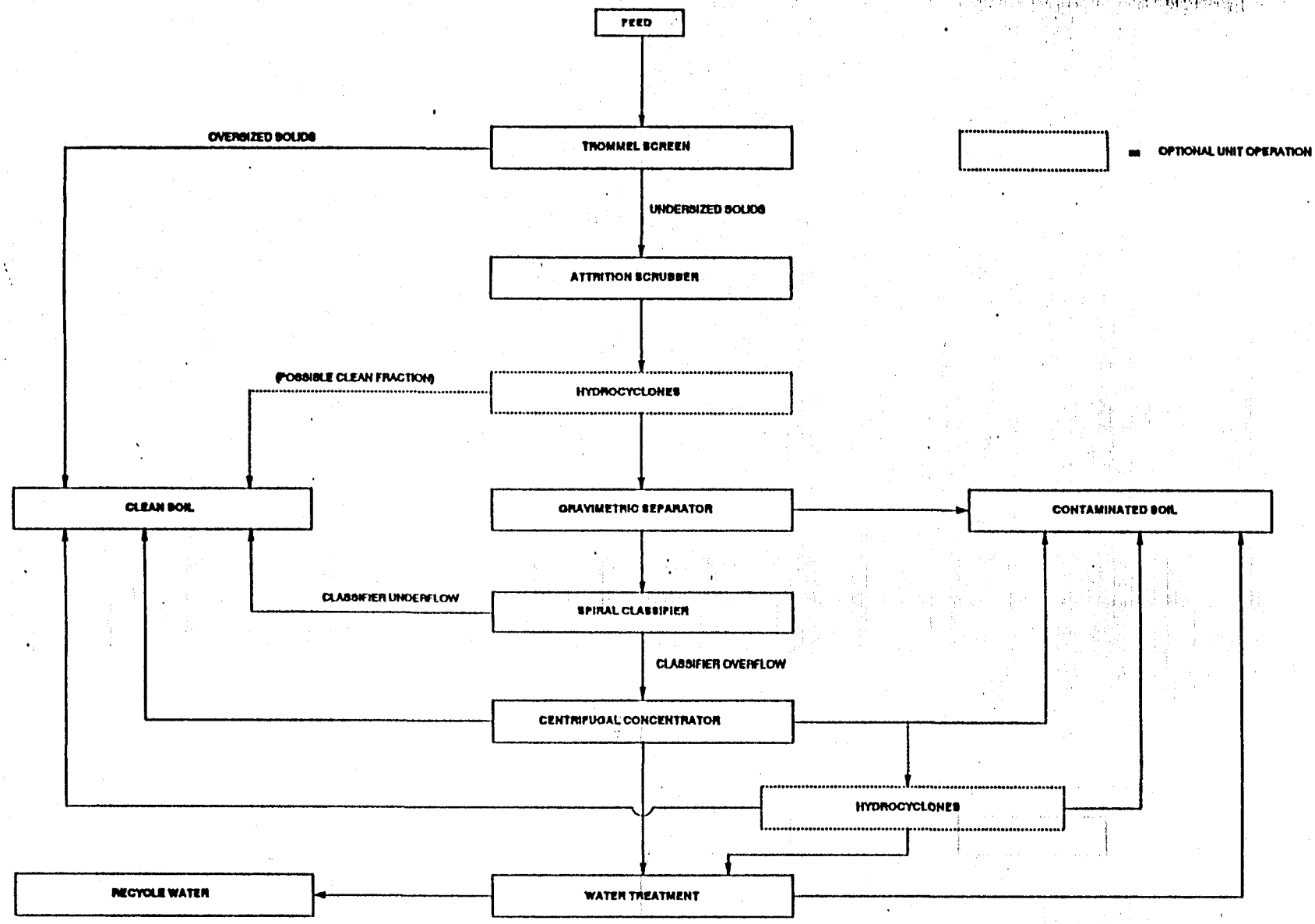


FIGURE 7.2-3
GENERAL FLOW DIAGRAM
TRUclean PROCESS

will be used in conjunction with the process equipment. LES&TC has provided the following SOPs. The SOPs are included in Attachment 2.

Process Equipment	SOP No.
Gravimetric Separator	SOP NTS 101
Centrifugal Gravimetric Separator	SOP NTS 102
Multi-Gravity Separator	SOP NTS 104
Trommel	SOP NTS 201
Attrition Scrubber	SOP NTS 203
Spiral Classifier	SOP NTS 301
Hydrocyclone Test Unit	SOP NTS 302
Thickener	

Support Equipment	SOP No.
Feed Auger	SOP NTS 202
Pump(s)	
Hopper(s)	
Tank(s)	
Homogenizer	
Agitator(s)	

7.3.1 Experimental Design

The experimental design includes two phases of test work. Phase I will consist of testing individual pieces of process equipment selected by LES&TC in order to determine the following:

- Effectiveness in separating clean soils from plutonium-contaminated soils
- Optimum size fraction(s) cleaned in the individual pieces of process equipment
- Optimum settings for the various physical variables for each piece of process equipment (e.g., incline angle and rpm)

- Optimum settings for the process variables for each piece of process equipment (e.g., gallons per minute (gpm) of wash water, pulp density, and feed rate).

Ten to fifteen individual samples of contaminated soil (approximately 22-23 kilogram each) will be charged to the piece of equipment to determine the optimum physical and operating parameters for a particular unit operation. There will be flexibility in the procedures to make adjustments for optimizing performance results. For example:

- If optimum conditions can be achieved with fewer tests on a particular piece of process equipment, a smaller number of tests will be conducted. If more tests are required, they will be conducted.

- If 22 to 23 kilograms is insufficient for optimization tests on a particular piece of equipment, more contaminated soils will be added to the test run. If 22 to 23 kilograms is an excess quantity, a smaller quantity will be used.

Once the Phase I data results are evaluated and the optimum design developed, and EG&G-RF or its designated contractor has approved the design, the Phase II portion of testing will begin. As part of Phase II, the process equipment units will be placed, based upon the Phase I evaluation, in the most effective sequence to separate clean soils from plutonium-contaminated soils. The experimental design for Phase II will consist of three runs (the Final Record Runs). Each of these runs will test 90 to 100 kilograms (approximately 200 lb.) of contaminated soils to effect the separation of clean soils from plutonium-contaminated soils.

7.3.2 Testing Facility

The TRUclean Process treatability tests will include testing of both the TRUclean Process and additional physical separation equipment. Tests will be performed at LES&TC testing facility.

7.3.3 Characterization of Test Soil

The RFP contaminated soils will be split for EG&G RFP, which will conduct chemical and radiological analysis. RFP characterization data will be compared with LES&TC characterization data. The characterization procedures discussed in this section are subject to change or modification.

LES&TC will generate a characterization sample from the RFP contaminated soils sent to them in four 55-gallon Department of Transportation (DOT) 17H drums. LES&TC will split the contaminated soil from the four drums, approximately 1450 kilograms (3200 lb.) by using the standard "cone and quartering" (Tyler, 1989) technique described below and shown in Figure 7.3-1:

> The sample will be piled in a cone, each shovelful going to the center of the cone and allowed to run down equally in all directions - this will mix the sample. Spread the pile out in a circle and walk around the pile gradually widening the circle with a shovel until the material is spread out to a uniform thickness. Rejected quaters will be stored separately and used based on the need during testing.
>
> Divide the flat pile into quarters and reject two opposite quarters. Mix again by shoveling the material into a conical pile, taking alternate shovelfuls from the two quarters saved. Continue the process

> of piling, flattening, and rejecting two quarters until the sample is reduced to the required size.
>
> Surveys have shown that the greatest cause of inconsistencies in testing results have come from improper selection of a sample which is not truly representative of the entire lot of material. Therefore, every care should be taken in the proper selection of the sample.

This procedure will be repeated five times to reduce the initial 1450 kilograms to approximately 90 to 100 kilograms (200 lb.). After the fifth repeat, the flattened pile will consist of four quarters of 22 to 23 kilograms. Opposite quarters will be combined resulting in two samples. One approximately 45 kilogram (100 lb.) sample, will be the LES&TC characterization sample (LES&TC-CS). The other, approximately the same weight, will be the RFP check sample. The RFP sample will be placed into a plastic bag, tie sealed, labeled, and placed into a drum dfor shipment to RFP together with residuals obtained during the LES&TC treatability tests.

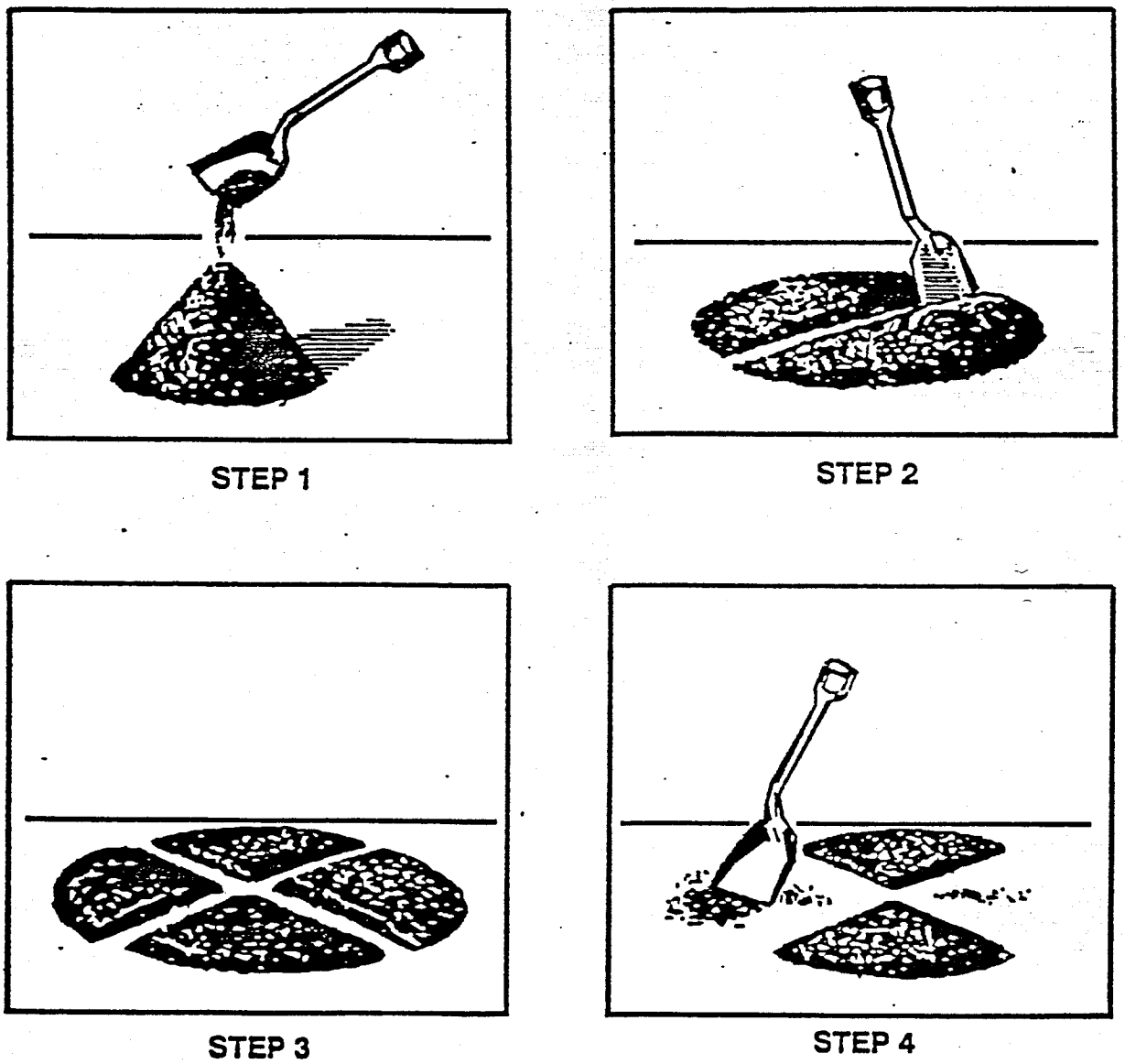


FIGURE 7.3-1
CONE AND QUARTERING

The approximate 45 kilogram LES&TC-CS will be characterized with the following tests. The sample will be air dried after the bulk density determination sample is taken and prior to the other characterization tests.

- Bulk density determination

Air-dry sample prior to performing the remaining tests

- pH determination
- Particle size analysis
- Air-dry moisture determination
- Gross alpha determination
- Gross beta determination
- Plutonium 239 plus 240 determination

7.3.3.1 Bulk Density Determination

The bulk density will be obtained in accordance with Analytical Procedures for Bulk Density Determination. The "volume filling" alternate should be used. The sample will be taken from the approximate 45 kilogram LES&TC-CS. The bulk density sample will then be returned to the full sample. The LES&TC Analytical Procedures are included in Attachment 2.

7.3.3.2 Air Drying

The approximate 45 kilogram LES&TC-CS will be air dried prior to the remaining characterization tests being conducted. The air drying procedure will be that described in Analytical Procedures for Air Dry Moisture Determination.

7.3.3.3 pH Determination

The pH determination will be obtained in accordance with Analytical Procedures for pH Determination. The sample will be a grab sample from the air dried LES&TC-CS.

7.3.3.4 Particle Size Analysis

The RFP contaminated soil sample could contain 0 to 5 percent plus 2-inch size cobbles, which will be hand picked at the sampling location. Total weight of these cobbles will be recorded for the future determination of acurate material balance,which will be prepared after performance of tests. The Particle Size Analysis will consist of three discrete screening ranges: plus 37.5 mm (+1.5-inch) to plus 19 mm (+3/4-inch), minus 19 mm (-3/4-inch) to plus 300 micron (+U.S. No. 50), and minus 300 micron to minus 45 micron (-U.S. No. 325).

1.5 inch - 3/4 inch

A Gilson Rocker Screen Set, SS-35, or equivalent, will be used to screen the entire LES&TC-CS, approximately 45 kilograms. The screen (sieve) sizes to be used are as follows:

millimeters	U.S. Sieve No.
37.5	1 1/2-in.
25	1-in.
19	3/4-in.

First, the 37.5 mm opening screen will be used in the Rocker Screen Set. The LES&TC-CS will be placed onto the screen, and the box rocked

to pass the minus 37.5 mm soils through the screen. Obvious clumps which are not coarse solid material will be manually broken to pass through the screen. The plus 37.5 mm material will be removed from the screen, weighed, and retained separately. The volume of the Rocker Screen Set box may require three repeats in order to screen/size the approximate 45 kilogram sample.

Second, the 25 mm opening screen will replace the 37.5 mm opening screen and the cycle will be repeated. The plus 25 mm material will be removed, weighed, and retained separately. The minus 25 mm material will be feed material to repeat the cycle using the 19 mm screen.

Once all of the LES&TC-CS has been dry screened through the sequence of sieve sizes to the 19 mm screen, the minus 19 mm contaminated soils will be set aside for later screening in the 19 mm to 300 micron sequence.

The separately retained size fractions will be subjected to wet screening in order to remove fine adhering particles. The procedure to be followed will be that outlined in Analytical Procedures, Particle Size Analysis - Wet Sieving Method. The screen oversize will be dried, weighed, and retained separately.

Those materials retained separately will be split using the following sequence, by size:

1) Place the material into a Gilson Model SP-1 sample splitter or equivalent

2) Adjust the chute opening width to a minimum of twice the particle size, preferably three times (e.g., for minus 37.5 mm use a 3-inch wide setting)

3) Each pass through the splitter will reduce the sample in half

4) Reduce the sample to the size required by LES&TC's internal laboratory procedures for chemical testing

5) When the final split size is selected, the other pan split in the sampler will be the RFP sample

6) Retain all the remaining samples from the pan splits as separate samples.

19mm - 300 micron

The minus 19 mm (-3/4-inch) contaminated soil from the dry screening and the oven dried fines from the wet screening will be combined. These soils will then be split in a Gilson Model SP-1 Sample Splitter or equal, down to approximately 4 kilograms (8.8 lb.). The sample will be split once more with half retained for the particle size analysis and half for the RFP sample. Retain all the remaining sample from the pan splits.

The approximate 2 kilograms (4.4 lb.) of LES&TC-CS particle size analysis sample fraction will be screened using the following sieve sizes:

millimeters(microns)	U.S. Sieve No.
9.5	3/8-in.
6.3	1/4-in.
4.0	5
2.0	10
(850)	20
(425)	40
(300)	50
PAN	

The particle size analysis will be obtained in accordance with Analytical Procedures, Particle Size Analysis, except that the above sieve sizes will be used. Both dry and wet sieving will be conducted.

The minus 300 micron (-U.S. No. 50) contaminated soil in the pan and the dried washed fines from wet sieving will be the feed material to the next set of sieve sizes. The following sizes will be used:

microns	U.S. Sieve No.
212	70
150	100
106	140
75	200
45	325
PAN	

The particle size analysis will be obtained in accordance with LES&TC Analytical Procedures, except that the above sieve sizes will be used.

7.3.3.5 Air-Dry Moisture Determination

The moisture determination will be obtained in accordance with Analytical Procedures - Air-Dry Moisture Determination. The sample will be obtained from the retained portion of the minus 19 mm, plus 300 micron split. This sample will be reduced to the size required by LES&TC Analytical Procedure by reducing the sample in the Gilson Model SP-1 Sample Splitter or equal.

7.3.3.6 Gross Alpha Determination

A gross alpha determination will be made on a sample of soil retained on every fraction (plus size) generated in the Particle Size Analysis as well as on the minus 45 micron sample. This will result in 20 determinations if particles are present in every sieve size.

The gross alpha determination will be made according to the LES&TC internal laboratory procedures in a manner sufficient to meet the requirements outlined in Section 5.2.

7.3.3.7 Gross Beta Determination

A gross beta determination will be made on a sample of soil retained on every fraction (plus size) generated in the Particle Size Analysis as well as on the minus 45 micron sample.

The gross beta determination will be made according to the LES&TC internal laboratory procedures in a manner sufficient to meet the requirements outlined in Section 5.2.

7.3.3.8 Plutonium 239 plus 240 Determination

A plutonium 239 plus 240 determination will be made on a sample of soil retained on every fraction (plus size) generated in the Particle Size Analysis as well as on the minus 45 micron sample.

The plutonium 239 plus 240 determination will be made according to the LES&TC internal laboratory procedures in a manner sufficient to meet the requirements outlined in Section 5.2.

7.3.4 Test Process

The TRUclean Process was specifically oriented to separate heavy contaminants from lighter non-contaminants. The initial development effort centered around the GS. Since the initial work, the TRUclean Process has grown into an assembly of physical scrubbing/classifying/separating pieces of equipment for segregating heavy contaminants from lighter non-contaminants.

Once the RFP sample has been received (55-gallon DOT 17H drums) and the LES&TC-CS removed, the treatability tests will begin. The treatability tests to be conducted on the RFP plutonium-contaminated soils will utilize most, if not all, of the process equipment listed in the introduction to Section 7.3. The steps in the treatability study are as follows:

1. Characterize the sample.

2. Test individual pieces of process equipment using water as the liquid medium.

3. Optimize the settings of the variables on the individual pieces of process equipment.

4. Test individual pieces of process equipment at the optimum settings using a sodium hydroxide solution at a pH of 12.0 to 12.5 as the liquid medium. The purpose of pH adjustment during testing is to identify the effect of different solution (besides water) for the reduction of Pu in soil.

5. Evaluate the performance results from the individual pieces of process equipment.

6. Assemble the selected pieces of process equipment into a sequential flow system that provides an expected optimum performance.

7. Test the optimum sequence system with water as the liquid medium.

8. Test the optimum sequence system with a sodium hydroxide solution at a pH of 12.0 to 12.5.

The initial parts of the test process are depicted on Figure 7.3-2, Feed Blending and Trommel Scrub/Screen. Several pieces of support equipment are shown on this figure. Once the LES&TC-CS has been removed as described in Section 7.3.3, the remaining sample will be coned and flattened once (Section 7.3.3, Figure 7.3-1). Samples for testing will be withdrawn from this disc shaped pile as required. Using a hoe or similar rabbling tool, a segment of contaminated soil will be withdrawn from the disc weighing approximately 23 kilograms (50 lb.). The sample will be withdrawn by placing the hoe at the center of the circle and withdrawing a thin segment. Additional samples for 23 kilogram tests will be withdrawn by selecting random radii and withdrawing soil from the center to the outer edge along the radii. For Phase I testing (Section 7.3.1), the sample size will be generally 23 kilograms; however, the sample size can be varied. For example, the trommel feed sample may need to be increased to 34 kilograms (75 lb.) in order to generate a 23 kilogram product for follow-on process equipment tests.

Prior to beginning the Phase II testing (Section 7.3.1), the remaining sample will be coned, flattened and either quartered or divided into thirds. This choice will depend on the amount of sample left. Phase II, the Final Record Runs, will consist of three runs, each consisting of approximately 90-100 kilograms of contaminated soil. The sample could be increased up to 114 kilograms (250 lb.) if a greater quantity of fine material is needed.

7.3.4.1 Support Equipment

There are several pieces of support equipment which will be used in association with most of the pieces of process equipment. These items include, but are not necessarily limited to the following: homogenizer

(Section 7.3.3), scale(s), feed hopper(s) with 3/4-inch opening screen deck, feed auger, collection containers, tank(s), and pump(s).

Scale(s):

All samples will be weighed prior to being tested and all products of the tests will be weighed. A small portion of every fifth feed sample will have its moisture content determined (AP, Air-Dry Moisture Determination).

Feed Hopper(s) with Screen Deck:

The RFP plutonium-contaminated soil sample is expected to contain particles larger than 3/4-inch. In order to protect the feed auger, the 3/4-inch material will be removed and saved in a container (Figure 7.3-2). The hopper's capacity is approximately five gallons (0.67 $ft.^3$).

Feed Auger:

The feed auger will be used to control the rate (kg/hr, lb/hr) at which the sample is added to the process equipment. The feed auger will be operated in accordance with LES&TC's Standard Operating Procedure, SOP NTS 202, Operation of feed auger. The Auger Feed Rate table, page 7 of 7, is based upon a density of 2.65 g/cm^3. The rheostat setting calibration will need to be recalculated based upon the LES&TC-CS bulk density determination. In general, the feed rate will be set to deliver a 23 kilogram sample to the process equipment in 10 minutes. This is equivalent to 138 kilograms (300 lb.) per hour. The feed rate will be adjusted higher or lower as the test results indicate it is appropriate.

Container(s):

Containers will be used to collect and accumulate the various products of each process equipment test during Phase I. During Phase

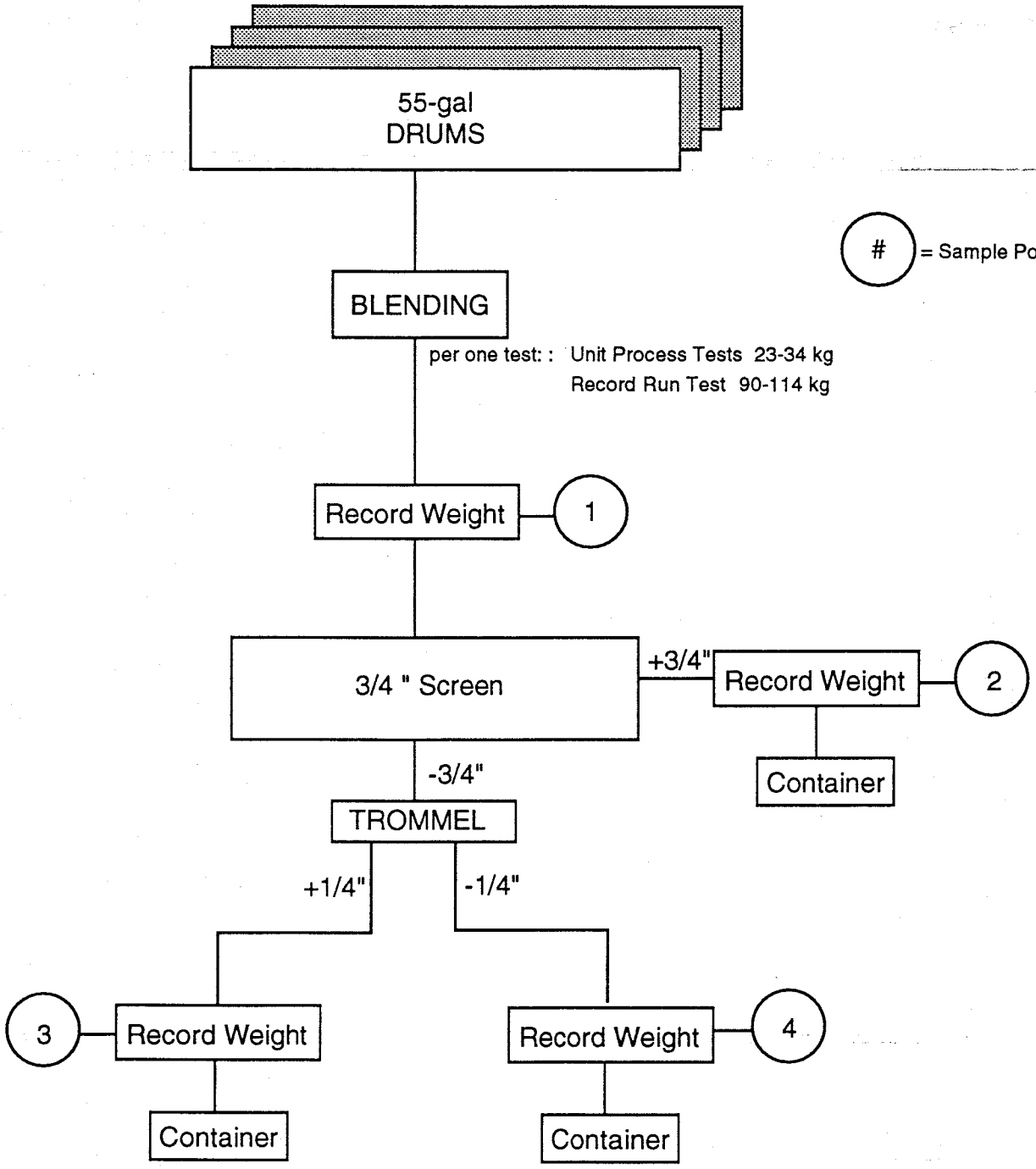


Figure 7.3-2. Blending and Trommel Scrub/Screen

II, process equipment units will be assembled in sequence. Containers will then be used for products at the end of the sequence.

Products which are collected can be dry solids (+3/4-inch material from the hopper screen), wet solids (screw classifier underflow), or slurries (hydrocyclone overflow). The containers will be tared prior to use and they will be weighed after use.

7.3.4.2 Process Equipment

The pieces of process equipment that will be tested as part of the treatability study program include the following: trommel, spiral classifier, GS, attrition scrubber, centrifugal gravity separator, multi-gravity separator (MGS), hydrocyclone test units, thickener, and filter(s).

LES&TC will have considerable flexibility in executing the treatability studies within the parameters of the TRUclean Process. This flexibility will include LES&TC's ability to choose items such as:

- Number of tests for each piece of process equipment
- Weight of soil for each test
- Setting of process equipment variables
- Sequence of testing process equipment
- Choice of process equipment to determine flow sheet configuration.

LES&TC will be required in the performance of the treatability tests to meet specific criteria, including but not necessarily limited to the following:

- Use water as the liquid medium to establish optimum operating conditions on each piece of process equipment to be tested.

- Use a sodium hydroxide solution at a pH range of 12.0 to 12.5 to test the process equipment at the optimum settings.

- Develop a plan for arranging the process equipment in sequence to generate the largest volume of clean (less than 0.9 pCi/g Plutonium 239 plus 240, Section 4.2) soils obtainable.

- Obtain approval of the plan from EG&G-RF or its designated subcontractor.

- Execute the plan at least three times using a minimum 91 kilograms (200 lb.) of contaminated soil and using a sodium hydroxide solution at a pH range of 12.0 to 12.5 at least once.

- Collect, record, analyze and report all data in accordance with the requirements outlined in Section 5.2.

- Conduct the six physical and chemical tests: particle size analysis, pH determination, air-dry moisture determination, gross alpha determination, gross beta determination, and plutonium 239 plus 240 determination, on every sieve size of every product generated.

- Conduct screen and sieve sizing to include: millimeters - 37.5, 25, 19, 9.5, 6.3, 4.0, and 2.0 (U.S. No. 5", 4", 3", 2", 1-1/2", 1", 3/4", 3/8", 1/4", 5, and 10); microns - 850, 425, 300, 212, 150, 106, 75, 45, and -45 (U.S. No. 20, 40, 50, 70, 100, 140, 200, 325, and -325) on every product generated as appropriate for the particle sizes present.

Descriptions of parameters for the process equipment of the TRUclean Process follow:

Trommel:

The trommel is an inclined rotating cylinder with internal spray bars and a perforated (1/4-inch openings) discharge tube. The rotation causes the larger soil particles to roll and tumble. This action removes fine particles adhering to larger ones. The water sprays wash the fines off the coarser particles (+ 1/4-inch, 6.3 mm). The trommel is shown in Figure 7.3-2 and its operation is covered by Standard Operating Procedure, SOP NTS 201. The trommel has four variables which can be adjusted to affect its operation: angle of tilt, feed rate,

spray bar water flow, discharge screen opening size. The initial settings will be:

Angle of tilt: The trommel will begin at zero setting which is approximately minus 10 degrees slope from horizontal. To obtain greater retention time, which will increase the scrubbing/washing time, raise the discharge end of the trommel (decrease the minus 10 degree slope to a lower negative slope).

Feed rate: The feed rate will be controlled by the feed auger (Section 7.3.4.2). The initial feed rate will always be set to deliver a 23 kilogram (50 lb.) test sample in 10 minutes. Adjustments for ensuing tests will be made as required.

For plus 3/4-inch contaminated soils the feed auger will be by-passed. The feed rate will be controlled by the size of test sample and frequency of addition when by-passing the feed auger.

Spray bar water flow: The initial spray bar water flow will always be set to deliver five gpm water. Adjustments for ensuing tests will be made as required. In general, the target pulp density inside the trommel is five to ten percent solids.

Discharge screen opening size: The discharge screen with the 1/4-inch (6.3 mm) openings will be used for all trommel tests.

Spiral Classifier:

Based upon the precept that the soil contaminants are small, heavy particles mixed with or adhering to larger clean soil particles, the spiral classifier is anticipated to be the optimum second step in the contaminated soil cleaning sequence. The spiral classifier can separate coarse particles (+150 micron, U.S. No. 100) particles from finer particles (-150 micron) by controlling spiral rpm, weir depth, and pulp density. The spiral classifier is shown in Figure 7.3-3 and its operation is covered by Standard Operating Procedure - Operation of the Spiral Classifier. The spiral classifier has four variables which can be adjusted to affect its operation: auger speed (screw rpm), feed rate, pulp density, and weir height. The initial settings will be set to achieve a split of minus 1/4-inch, plus 70 U.S. No. sieve (-6.3mm, +212 microns) in the underflow. This size split is chosen as the initial starting

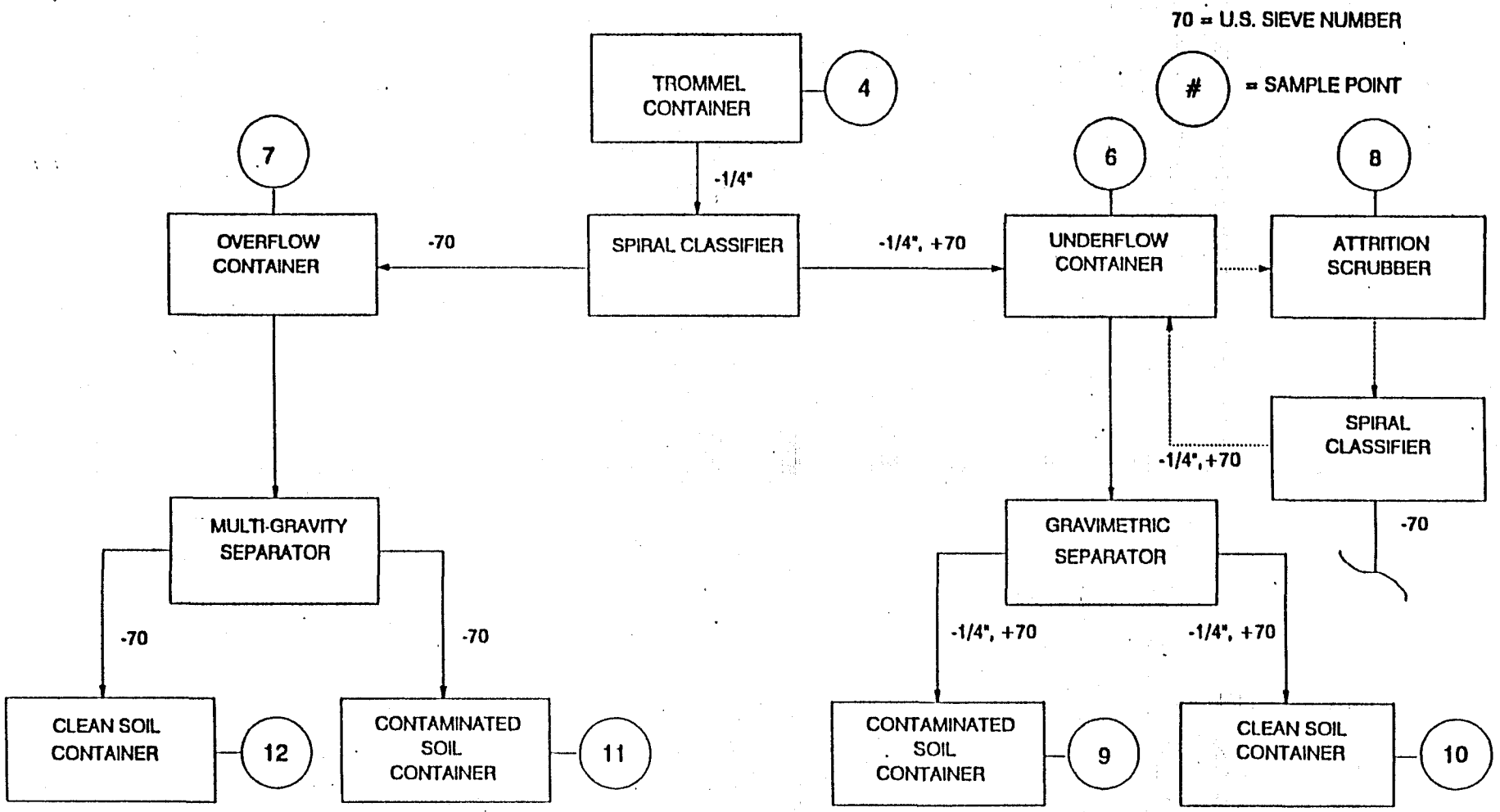


FIGURE 7.3-3
CLASSIFIER SIZING
AND GRAVITY SEPARATION

point because if the particles do not meet the "clean" requirements, they are in the preferred size range for the GS and/or the attrition scrubber.

Auger speed: the auger speed will be set at 25 rpm for the initial test. Adjustments will be made to achieve a split between underflow and overflow at U.S. Sieve No. 70.

Feed rate: The feed rate will be controlled to deliver a 23 kilogram test sample in 10 minutes. Adjustments for ensuing tests will be made as required.

Pulp density: The pulp density will be adjusted as required to achieve the desired split at U.S. Sieve No. 70. Water sprays will be used to wash fine particles off of the coarse particles being raised by the spiral (underflow). This volume of water will impact the pulp density.

Weir height: The initial weir height will be set at the maximum height. Adjustments will be made as required to achieve the desired size split.

The spiral classifier underflow, Figure 7.3-3, item 6 will be sampled in order to conduct the six physical and chemical tests discussed in Section 7.3.6. These tests will indicate the effectiveness of the separation of contaminants from the clean soil. If the spiral classifier underflow is not "clean", further treatment and/or adjustments to the split size will be required. The underflow can be further treated by the GS or attrition scrubber.

Attrition Scrubber:

The attrition scrubber in shown in Figure 7.3-3 and its operation is covered by Standard Operating Procedure, SOP NTS 203. The attrition scrubber is an agitation device in which particles continually abrade each other. This removes fine (-150 micron) particles from larger (500 to 150 microns) particles. The attrition scrubber has four variables which can be adjusted to affect its operation: pulp density, impeller speed, feed rate, and residence time. The attrition scrubber is a two cell unit consisting of two approximately 1 $ft.^3$ cells. The attrition scrubber operates in series with the first cell, discharging to the second. LES&TC may have available a new two cell unit for the

treatability tests. The new unit's cells will operate in series and be smaller, approximately 0.33 $ft.^3$ to 0.5 $ft.^3$ cells.

Pulp density: The pulp density will be set for 70 percent solids for the initial test. Adjustments will be made as required to effect better scrubbing.

Impeller speed: The initial impeller speed will be set according to the manufacturer's recommendation.

Feed rate: The feed rate will be controlled to deliver a 23 kilogram test sample in 10 minutes. Adjustments for ensuing tests will be made as required. Feed rate affects retention time.

Residence time: The longer the residence time, the greater the scrubbing contact. Residence times are adjustable from 3 minutes to 30 minutes. After the initial feed rate setting, adjustments to residence time will be made as required.

A sample, Figure 7.3-3, item 8, will be obtained from the attrition scrubber in a manner similar to that described for the minus 1/4-inch fraction (-6.3 mm) in Section 7.3.6. The results of the six physical and chemical tests will indicate the effectiveness of the scrubbing and the sieve size to achieve a separation of "clean" and contaminated soils. It is anticipated that the attrition scrubber product will be treated best by a return to the spiral classifier.

Gravimetric Separator:

The GS is shown in Figure 7.3-3 and its operation is covered by Standard Operating Procedure, SOP NTS 101. The GS separates heavy (>7.0 Sp. Gr.) particles from lighter particles due to the faster settling rate of the heavy particles in a pulse of water. The GS has six variables which can be adjusted to affect its operation: stroke length, stroke frequency, bedding material, water flow rate, feed dilution water and feed rate. The GS operates best in the particle size range of minus 3/8-inch, plus U.S. No. 200 (-9.5 mm, +75 micron).

Stroke length: Stroke length will be set at 1/2-inch for the initial test. Adjustments will be made as required.

Stroke frequency: The stroke frequency will be set a 125 strokes per minute for the initial test. Adjustments will be made as required.

Bedding material: The initial bedding material will consist of 3/16-inch steel balls placed to a thickness of 1-inch on the support screen. The support screen openings are fixed at 1/8-inch.

Water flow rate: The initial water flow rate will be set at 5 gpm. Adjustments will be made as required.

Feed dilution water: The initial feed dilution water will be set to produce a 30 percent solids by weight pulp density. Adjustments will be made as required.

Feed rate: The feed rate will be controlled to deliver a 23 kilogram test sample in 10 minutes. Adjustments for ensuing tests will be made as required.

Samples to determine the effectiveness of the GS will be taken from both the anticipated "clean" container (overflow) and the contaminated soil container(s) (hutch and bed). Samples will be obtained and analyzed as discussed in Section 7.3.6. As with all AWC analyses, a split sample will be taken for RFP.

Multi-Gravity Separator:

The MGS is shown in Figure 7.3-3 and its operation is covered by Standard Operating Procedure, SOP NTS 104. The MGS separates heavy (>4.0 Sp. Gr.) particles from lighter particles via a combination of centrifugal and velocity forces, (e.g., rotational speed and shaking frequency). The MGS has seven variables which can be adjusted to affect its operation: rotational speed, shake frequency, amplitude, washwater flow rate, angle of tilt, feed rate, and pulp density. The MGS operates best on liberated contaminants in the minus 250 micron (-60 U.S. No.), plus 1 micron range. The spiral classifier overflow will be targeted to be minus 250 microns which makes the MGS an appropriate piece of process equipment to test on spiral classifier overflow.

Rotational speed: The rotational speed will be set a 200 rpm for the initial test. Adjustments will be made as required. Rotational speed settings are 160, 200, or 240 rpm.

Shake frequency: The shake frequency will be set at 4.8 cycles per second (cps) for the initial test. Adjustments will be made as required. Shake frequency settings are 4.0, 4.8, or 5.7 cps.

Amplitude: The amplitude will be set at 19.0 mm for the initial test. Adjustments will be made as required. Amplitude settings are 12.7, 19.0, or 25.4 mm.

Washwater flow rate: The washwater flow rate will be set at 5 liters per minute (lpm) for the initial test. Adjustments will be made as required. Flow rates are adjustable from 0 to 10 lpm.

Angle of tilt: The tilt angle will be set at 2.5 degrees for the initial test. Adjustments will be made as required. Tilt angles are adjustable from 0 to 5 degrees.

Feed rate: the feed rate will be controlled to deliver a 23 kilogram test sample in 10 minutes. Adjustments for ensuing tests will be made as required.

Pulp density: The pulp density will be set at 30 percent solids by weight for the initial test. Adjustments will be made as required. Pulp density is adjustable from 15 to 50 percent.

Samples to determine the effectiveness of the MGS will be taken from three locations, items 7, 11, and 12 as shown on Figure 7.3-3. Samples will be obtained and analyzed as discussed in Section 7.3.6. As with all samples taken for LES&TC analyses, a split will be taken for RFP.

Hydrocyclone:

The hydrocyclone is shown in Figure 7.3-4 and its operation is covered by Standard Operating Procedure, SOP NTS 302. The hydrocyclone separates different particle sizes into a plus and minus size fractions. Hydrocyclones can make good separations over wide particle size ranges. For example, hydrocyclones can separate particles at plus and minus 1 millimeter or plus and minus 75 microns. The hydrocyclone has four variables which can be adjusted to affect its operation: pulp density, inlet pressure, vortex diameter, and unit size. The hydrocyclone has the capability to split the very fine particles away from coarser particles. The hydrocyclone will be tested to determine two items:

1) capability to separate contaminated soils from "clean" soils, items 13 and 14, respectively on Figure 7.3-4, and 2) capability to provide improved sized feed to the centrifugal gravity separator and the MGS.

Pulp density: The pulp density will be set at the optimum spiral classifier pulp density for the initial test. Adjustments will be made as required.

Inlet pressure: The inlet pressure will be set at 75 psi for the initial test. Adjustments will be made as required. Inlet pressure is adjustable from 50 to 100 psi.

Vortex diameter: The vortex diameter for the initial test will be selected based upon the hydrocyclone manufacturer's recommendation to control the underflow at minus 70 U.S. No., plus 200 U.S. No. with 5 percent or less (preferred) minus 200 U.S. No. particles in the underflow.

Unit size: For the particle size separations discussed, the 10 mm hydrocyclone(s) will be used.

The hydrocyclone overflow and underflow will be sampled and analyzed, items 13 and 14, Figure 7.3-4. Tests will be conducted to try to achieve a "clean" underflow. If a clean underflow is not achieved, the underflow will be tested in the centrifugal gravity separator. In either case the hydrocyclone overflow will be tested in the MGS as described for treating spiral classifier Overflow.

Centrifugal Gravity Separator:

The centrifugal gravity separator (CGS) is shown in Figure 7.3-4 and its operation is covered by Standard Operating Procedure, SOP NTS 102. The CGS separates heavy (>4.0 Sp. Gr.) particles from lighter particles via a combination of centrifugal and velocity forces. The CGS works better on particles smaller than 212 microns. The CGS has five variables which can be adjusted to affect its operation: feed dilution, feed size, feed rate, bowl back pressure, and shaking screen vibration (frequency and amplitude).

Feed dilution: The feed dilution will be set at 40 percent solids by weight for the initial test. Adjustments will be made as required. The minimum feed dilution is 30 percent solids.

Feed size: The feed size will be set at minus 70 U.S. No., plus 200 U.S. No. (-212, +75 microns) for the initial test. Adjustments will be made as required.

Feed rate: The feed rate will be controlled to deliver a 23 kilogram test sample in 10 minutes. Adjustments for ensuing tests will be made as required.

Bowl back pressure: The bowl back pressure will be set at 9 psi for the initial test. Adjustments will be made as required. The bowl back pressure is adjustable from 0 to 30 psi. For minus 100 U.S. No. (-150 micron), normal pressures are in the 4 to 6 psi range.

Shaking screen vibration: The shaking screen vibration, frequency and amplitude, will be set at the mid-range setting for each one for the initial test. Adjustments will be made as required.

Samples to determine the effectiveness of CGS will be taken from three locations, items 14, 15 and 16 as shown on Figure 7.3-4. Samples will be obtained and analyzed as discussed in Section 7.3.6. As with all samples taken for LES&TC analyses, a split will be taken for RFP.

Thickener:

A center-well-feed-thickener will be used to test the recovery of scrub/wash/dilution water for recycle. Flocculants and pH adjustments will be tested to obtain the cleanest possible thickener overflow. It is critical that recycle water contain low enough levels of radionuclides so that they contribute less than half, ideally one-tenth, the radionuclide levels set in the "clean" definition before being recycled.

7.3.5 Equipment, Materials and Reagents

The major pieces of equipment that will be utilized are in three areas: Process Equipment, Support Equipment, and Sampling/Analytical Equipment.

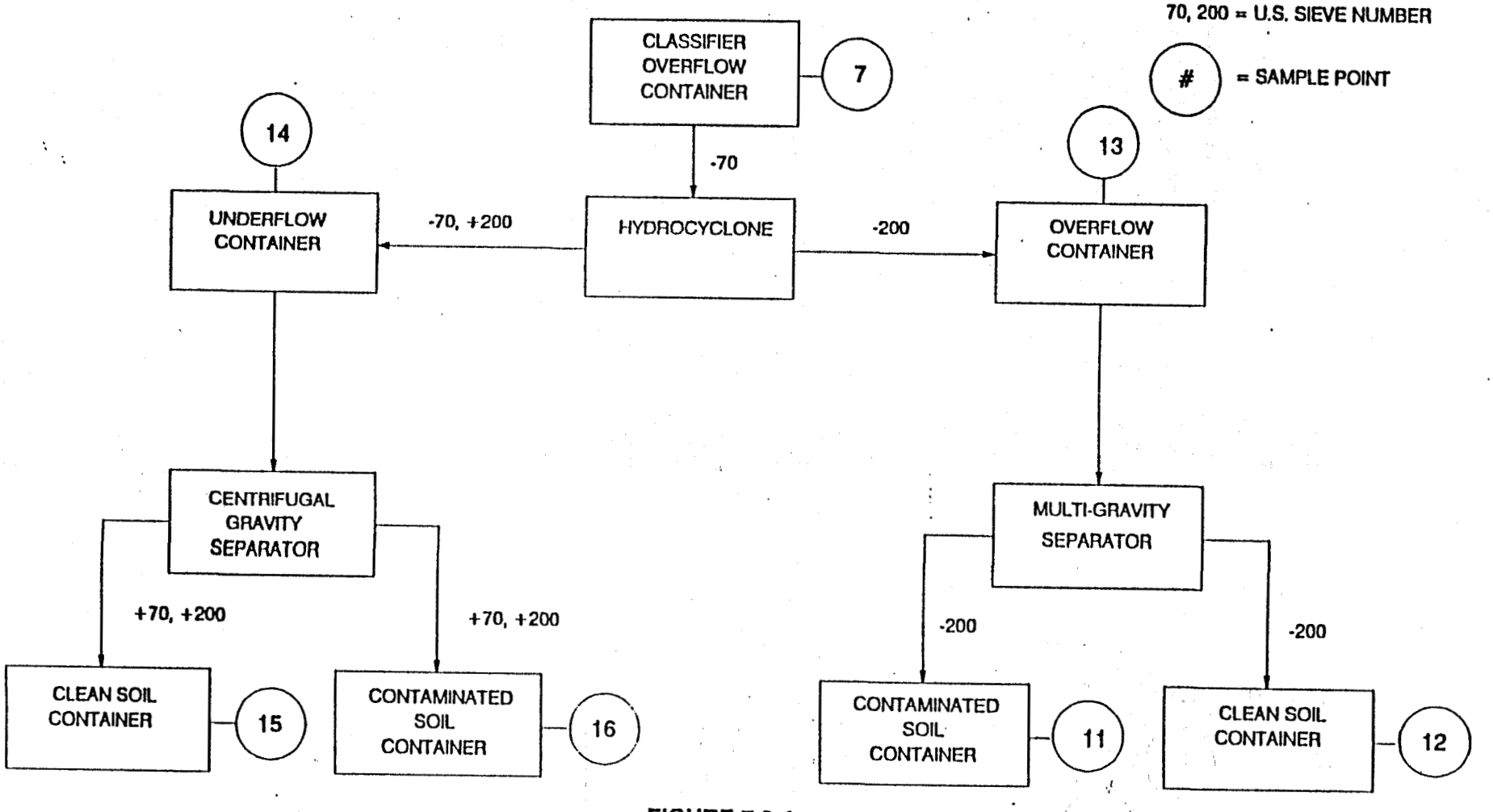


FIGURE 7.3-4
HYDROCYCLONE SIZING
AND GRAVITY SEPARATION

7.3.5.1 Process Equipment

- Trommel
- Spiral Classifier
- Attrition Scrubber(s)
- Gravimetric Separator
- Hydrocyclones
- Centrifugal Gravity Separator
- Multi-Gravity Separator
- Thickener

7.3.5.2 Support Equipment

- Feed Auger(s)
- Hopper(s) with screen
- Containers: tanks, drums
- Pumps
- Agitators
- Filter(s)
- Scale(s)

7.3.5.3 Sampling/Analytical Equipment

- Homogenizer
- Gilson, Rocker Screen Set, SS-35 or equivalent; U.S. No. Screens: 1 1/2-, 1-, and 3/4-inch
- Gilson Model SP-1, Sample Splitter or equal with three pans, brushes
- Tyler Sample Reducer (16:1) or equivalent with three pans, brushes
- Hoe or rabble

- Laboratory sieve screen sets to cover sizes U.S. No. (mm or microns):

  3/8-inch (9.5 mm)
  1/4-inch (6.3 mm)
  U.S. No.5 (4.0 mm)
  U.S. No. 10 (2.0 mm)
  U.S. No. 20 (850 micron)
  U.S. No. 40 (425 micron)
  U.S. No. 50 (300 micron)
  U.S. No. 70 (212 micron)
  U.S. No. 100 (150 micron)

U.S. No. 140	(106 micron)
U.S. No. 200	(75 micron)
U.S. No. 325	(45 micron)
PANS	

- Analytical equipment for pH, gross alpha, gross beta, and plutonium 239 plus 240 (gamma/alpha spectrometer)

7.3.5.4 Reagents

The only reagent that will be used as a treatability testing media will be sodium hydroxide. Sodium hydroxide will be added to the water to achieve a solution pH of 12.0 to 12.5 which will be used in the process equipment for scrubbing, washing, and pulp density control. Flocculants may be tested in the thickener to improve settling and filtering characteristics.

7.3.6 Measurements and Sampling Requirements

As discussed in Section 7.3.4.1, containers will be used to collect and accumulate the various products of each process equipment test. Each of these products will be sized according to the sizes described in Section 7.3.3.4, Particle Size Analysis. Depending on the weight of the product, the product will be split to obtain a representative sample of less weight for physical and chemical testing to determine the effectiveness of the separation of contaminants from soil. For example, the plus 3/4-inch (+19 mm) product material will be placed into a Gilson Rocker Screen set, SS-35 or equal containing a 50 mm (U.S. No. 2-inch) screen. The plus 50 mm material and the minus 50 mm will be split as described in Section 7.3.3.4, 125 - 19 mm. If all size fractions are present, there will be seven fractions for which

physical and chemical data will be collected. In general, these are shown on Figure 7.3-2 as item 2 when the material is dry and as item 5 when the material is wet. The data to be collected includes:

- Moisture content if sample is from wet solids (Section 7.3.3.5)
- Particle size analysis (Section 7.3.3.4)
- pH determination (Section 7.3.3.3)
- Gross alpha determination (Section 7.3.3.6)
- Gross beta determination (Section 7.3.3.7)
- Plutonium 239 plus 240 determination (Section 7.3.3.8).

The minus 3/4-inch (-19 mm), plus 1/4-inch (+6.3 mm) fraction (Figure 7.3-2, Item 3) will be split down to the LES&TC standard sample size in the same manner as referred to above for the minus 50mm (-2-inch U.S. No.) and described in Section 7.3.3.4. This trommel product will be wet. The above six physical and chemical tests will be performed on each size fraction present consistent with the screen and sieve designations in Section 7.3.3.4.

During treatability testing, observation and judgement will be required in the handling of the minus 1/4-inch (-6.3mm) soils (Figure 7.3-2, Item 4). In general, if the minus 1/4-inch fraction is coarse, settling will occur quickly and the scrubbing/wash solutions will become clear enough to decant. When this occurs, the sample will be split down to the LES&TC standard sample size by use of a Tyler Sample Reducer (16:1 sample reducer) or equivalent. The Tyler Sample Reducer will be especially helpful in splitting samples from the spiral classifier underflow (Figure 7.3-3, Item 6). The sample reducer is limited to a maximum particle size of 1/2-inch (12.5mm). Once the proper

sample is obtained, the above mentioned six physical and chemical tests will be performed on each sieve fraction present consistent with Section 7.3.3.4.

If the minus 1/4-inch fraction is primarily fine soils, then settling will be slow. In order to collect the sample in this circumstance the container contents will be agitated at high speed with a portable agitator for at least 15 minutes. The agitated container contents then will be poured into the 16:1 sample reducer to obtain the sample. The sample will be subjected to the six physical and chemical tests described above and consistent with the sieve separations in Section 7.3.3.4.

Decant solutions and suspensions of fine solids will be put into the thickener for settling characteristic testing. A split of all samples will be taken and retained for RFP analysis.

All measurements and determinations will be made as outlined in Section 7.3 subsections and according to LES&TC internal laboratory procedures in a manner sufficient to meet the requirements outlined in Section 5.2. Where LES&TC internal procedures and work plan directions conflict, the work plan will take precedence unless resolved in writing by EG&G-RF or its designated subcontractor.

7.4 Field and Laboratory Documentation and Management

Management of the QA Records and data generated in support of the TRUclean treatability test is addressed in the Quality Assurance Project Plan for Land Surface Cleanup of Plutonium Project (LES&TC) and LES&TC SOPs included in Attachment. Records of the operating conditions for the test equipment during treatability testing include project logbooks and equipment specific run sheets.

LES&TC deliverables at the completion of the treatability tests include the following:

- performance evaluation report
  - project logbook containing results of physical and chemical tests, weights, temperatures, and other pertinent information.
  - process equipment run sheets.
- originals (or legible copies) of all QA Records produced throughout the treatability testing, organized by test run number (e.g., 1 through 5).
- test parameters and analytical results in Lotus format, organized by test run number.

The EG&G-RF project manager will receive the deliverables supporting the treatability test from the test subcontractor. The project manager will manage these data according to the EM data management procedures which are presently being developed. It is noted that some technical data specifications and documents are considered proprietary to LES&TC and will be handled accordingly. EG&G-RF will provide the test data and results to their feasibility study subcontractor for interpretation and analysis. The subcontractor is required to manage these data in accordance with a standardized document inventory and filing system. Data interpretation and analysis results will be documented to allow independent verification of the conclusions drawn.

7.5 Data Evaluation

Data evaluation will be a critical component of the TRUclean Process treatability test program. Data evaluation will be performed on the physical and chemical results from three areas: LES&TC-CS, individual process

equipment tests (Phase I), and sequential process equipment tests (Phase II). After all the treatability tests are complete and the data evaluated, an overall project data evaluation will be made.

7.5.1 LES&TC Characterization Sample

Prior to individual process equipment testing being undertaken, the LES&TC-CS data will be reviewed and evaluated. Since the objective of the treatability test program is the separation of contaminated soils into clean (less than 0.9 pCi/g plutonium 239 plus 240, Section 4.2) and contaminated soils, evaluation of the contaminated soil starting material is a key element.

The items to be evaluated will include, but are not necessarily limited to, bulk density, moisture content, pH, particle size analysis, gross alpha, gross beta, and plutonium 239 plus 240. Each plus screen fraction and the minus 45 micron (-325 U.S. No.) fraction data will be reviewed. If all particle sizes are present (Section 7.3.3.4) each of them will be evaluated. Data will be presented in a tabular format (e.g., tests as column titles and screen fractions as the vertical column items).

The data will be reviewed with the prime effort being expended on the alpha, beta, and plutonium concentrations of each size fraction. Comparisons will be made between activities of dry screened soils and wet screened soils. These differences will be indicative of the adherence of fine contaminated particles to larger clean soil particles. Evaluations will determine whether the "clean" condition is achieved.

These evaluations will be starting point for the individual process equipment tests. For example, evaluation of the data will indicate whether

the plus 3/4-inch soils will be clean and not need to be treatability tested, or whether they will be contaminated and will need to be treatability tested.

7.5.2 Individual Process Equipment Tests

Individual pieces of process equipment will be tested to determine the equipment's effectiveness in separating clean soils from contaminated soils. In addition to the items to be evaluated as described for the LES&TC-CS, a material balance around each piece of process equipment for each solution and solid streams will be required. To accomplish the material balance, weights and moisture contents become important data along with the alpha, beta, and plutonium determinations.

The trommel feed will be based upon the LES&TC-CS results. Trommel feed will be characterized prior to processing and material balance will be calculated after the test.

In general, the trommel products, plus 1/4-inch soils and minus 1/4-inch soils will be characterized sufficiently well to use that characterization data as the feed data for the next piece of process equipment. For each piece of process equipment, data on that piece of equipment's variables will be collected, recorded, tabulated, and compared to see the impact on the physical and chemical data. For example, if the initial trommel settings do not produce a clean plus 1/4-inch soil, the data will be evaluated by reviewing the screen fractions to determine:

1. Plus 1/4-inch particles that are still contaminated

2. Plus 1/4-inch particles that are contaminated by minus 1/4-inch carry-over with the plus 1/4-inch soils.

Based upon this type of data evaluation it may be determined that trommel variables should be changed, one at a time. For example, the incline angle may be reduced to provide longer retention time in the perforated discharge tube to allow better washing of the plus 1/4-inch particles to remove fines.

Evaluations will be made for each piece of process equipment by tabulating the characterization type data and comparing it against different variable settings for that piece of process equipment. A written description will be provided to record the interpretation of the data and the rationale for the change in the process equipment variable(s), and the results of the change.

When the best results are achieved using a water media, then one or more tests will be run using a water/sodium hydroxide solution at pH 12.0 to 12.5. The high pH solution results will be compared to the water only results.

Once all the pieces of process equipment chosen by LES&TC for treatability testing have been tested and evaluated, LES&TC will develop a plan for the sequence testing of process equipment, Phase II. The Phase II plan will be supported by summarizing collected data from individual tests. The plan will be presented to EG&G-RF or their designated subcontractor for approval.

7.5.3 Sequential Process Equipment Tests

Once the sequence of process equipment has been approved, this portion of testing can begin. During this Phase II portion of the treatability testing, the process equipment variables will not be changed. Their optimum setting will have been determined by Phase I testing.

The data collected will be as delineated in the previous two sections. This data will encompass the trommel feed and the final products of the TRUclean Process unit operations. This data will be evaluated to provide input for the overall TRUclean Process evaluation. For example, spiral classifier underflow, which is expected to be clean, would be a final product. Spiral classifier overflow, which will be contaminated is not a final product. The overflow is fed to another piece of process equipment, as such, its characterization will not be part of the sequential process equipment evaluation.

Samples will be collected and characterized at all process equipment discharge points. The data from those intermediate discharge points, although not part of the final data evaluation, will be retained to interpret what occurred if the sequential systems cannot produce either clean soil or a sufficient quantity of clean soil.

7.5.4 Overall Data Evaluation

The overall data evaluation will follow the same procedures as described earlier. Figure 7.5-1 is presented as a hypothetical example of the data evaluation procedure which is the input to the decision process.

7.6 Regulatory Requirements

Off-site treatability studies on RFP plutonium-contaminated soil require conformance to applicable DOT regulations. These regulations address the handling and shipping requirements for radioactive materials. It is expected that the soil will be exempt from the requirements of 40 CFR 261 - Identification and Listing of Hazardous Waste; therefore, the soil will not

be subject to the requirements of 40 CFR 262 - Standards Applicable to Generators of Hazardous Waste.

The TRUclean Process tests on RFP plutonium-contaminated soil will be performed at the LES&TC testing laboratory. Determination of shipment requirements for the bulk soil sample will be made as outlined in the Attachment 1 of this work plan.

The weight limitation of treated soil for Type A packages returning to the RFP from the NTS is estimated under the assumption that the TRUclean Process produces a "clean" soil stream that meets the cleanup goal of 0.9 pCi/g plutonium and comprises 90 percent of the original feed mass for the entire RFP bulk soil sample. This assumption leads to a material balance around the TRUclean Process as represented in Figure 7.6-1.

7.7 Residuals Management

It is anticipated that at the end of the test program there will be the following residuals:

1) RFP soils, unused

2) Dry solids including: a) "clean" soils (Pu 239 plus 240 less than 0.9 pCi/g), b) contaminated soils with Pu 239 and 240 in concentrations equal to or less than the original RFP soils, and c) contaminated soils with Pu 239 and 240 in concentrations higher than the original RFP soils.

3) Wet solids including: a) "clean" soils (Pu 239 plus 240 less than 0.9 pCi/g), b) contaminated soils with Pu 239 and 240 in concentrations equal to or less than the original RFP soils, and c) contaminated soils with Pu 239 and 240 in concentrations higher than the original RFP soils.

4) Sludges/slurries including: a) "clean" soils (Pu 239 plus 240 less than 0.9 pCi/g), b) contaminated soils with Pu 239 and 240 in concentrations equal to or less than the original RFP soils, and c) contaminated soils with Pu 239 and 240 in concentrations higher than the original RFP soils.

5) Water solutions including: a) solutions sufficiently clean to be disposed of within LES&TC's permit for non-hazardous/non-radioactive liquid disposal, b) contaminated solution not requiring radioactive placarding for shipment under DOT 49 CFR regulations, c) contaminated solutions requiring placarding under DOT 49 CFR.

6) Sodium Hydroxide solutions including: a) solutions "radioactively" clean and neutralized to be disposed of within LES&TC's permit for non-hazardous/non-radioactive liquid disposal, b) contaminated solutions not requiring radioactive placarding and neutralized for shipment under DOT 49 CFR, c) contaminated solutions requiring radioactive placarding and high pH classification under DOT 49 CFR.

All materials and solutions, except items 5.a and 6.a, will be returned to the RFP. All materials and solutions in items 1, 2.a, 2.b, 3.a, 3.b, 4.a, 4.b, 5.b, and 6.b will be returned to the RFP in DOT 17H 55-gallon drums. All materials and solutions in items 2.c, 3.c, 4.c, 5.c, and 6.c will be returned to RFP in DOT-specified and approved drums based upon the criteria set forth in 49 CFR.

LES&TC will be responsible for preparing all residuals for return shipment to RFP. EG&G-RF will be responsible for the handling and disposition of residuals upon receipt at RFP.

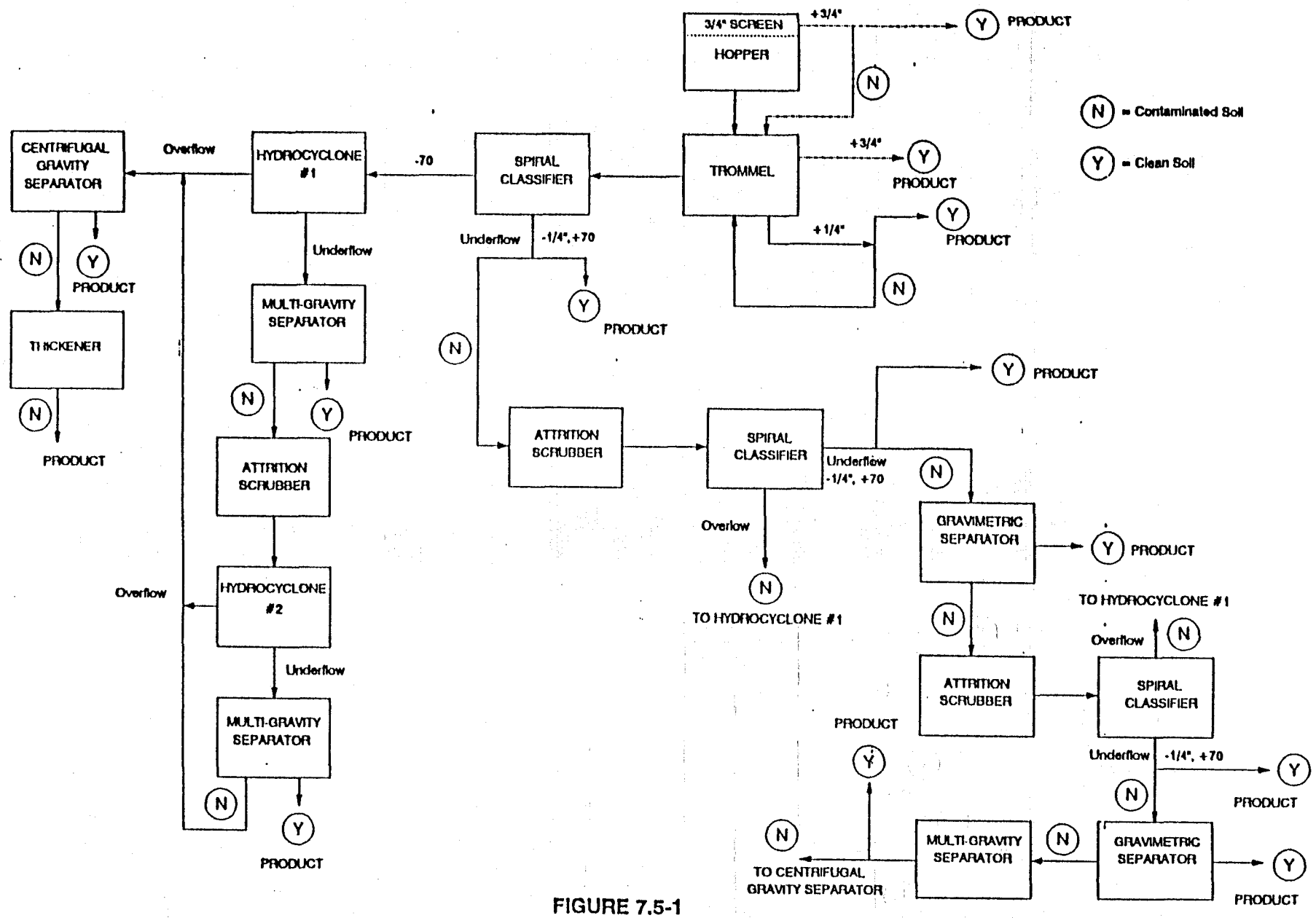


FIGURE 7.5-1
HYPOTHETICAL DECISION TREE
TRUclean PROCESS

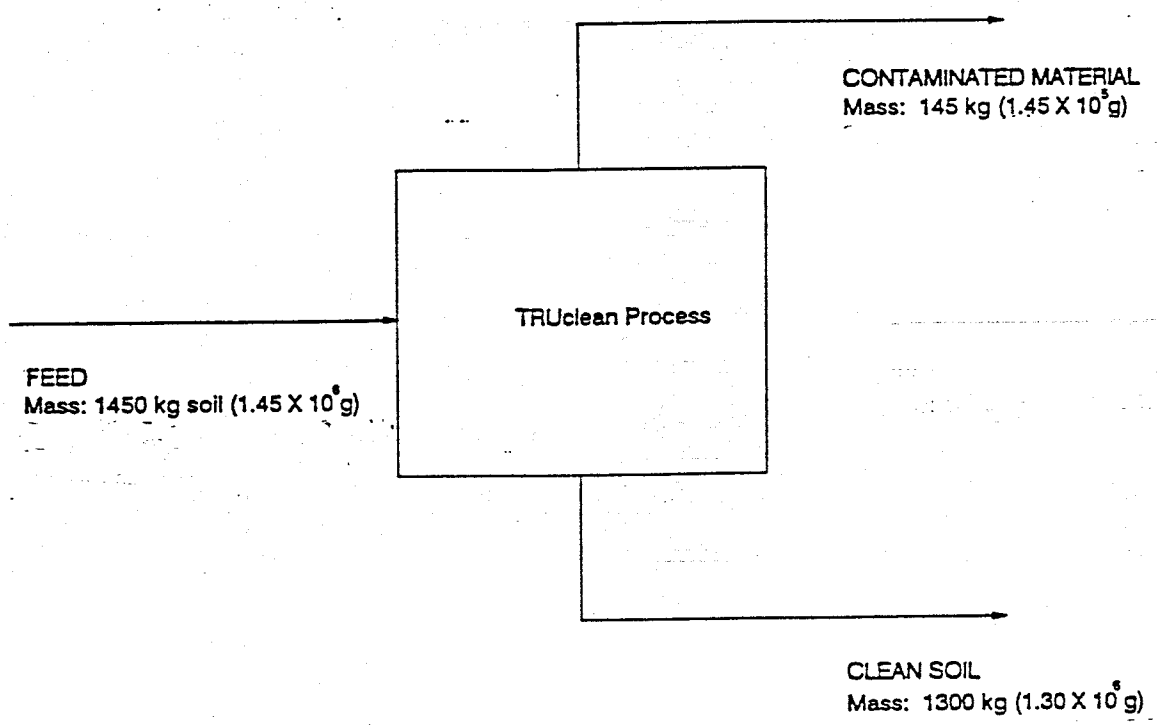


FIGURE 7.6-1

ASSUMED OVERALL MATERIAL BALANCE

TRUclean PROCESS

7.8 Health and Safety

Activities conducted at the LES&TC facility will be performed in accordance with the Environment Safety and Health Plan for the Land Surface Clean-Up of Plutonium Project (LSCOPP) dated April 22, 1991. Specific procedures have been established for nonradiological and radiological hazards. These procedures, as referenced above, have been written in accordance with the applicable DOE Orders.

A complete set of the applicable EG&G-RF/EM procedures should be reviewed by the operators. Particular attention should be applied to the following areas:

- Radioactive Airborne Contamination Monitoring
- Contamination Surveys - Personnel and Equipment
- Internal/External Dosimetry Requirements
- Decontamination and Equipment Release Surveys
- Personnel Training in Radiation Safety and Contamination Reduction Techniques
- Nonradiological Hazards; i.e., electrical, mechanical, fire.

The primary hazard is from the low levels of plutonium that will be concentrated, increasing the exposure potential to the worker. It should be noted that ingestion and inhalation of plutonium contaminated soil are dangers associated with implementation of TRUclean process testing. Through the proper execution of the procedures listed in the ES&H LSCOPP Plan no additional exposure is anticipated.

7.9 Schedule for TRUclean Tests

A bar chart schedule of the TRUclean treatability tests is included as Figure 7.9-1.

ITEM	COMPLETION DATE
Ship soil samples to LES&TC	March, 1993
Perform Treatability Study	June, 1993
Draft Treatability Study Report	June, 1993
Receive all Analytical Data	August, 1993
Final Treatability Study Report	September, 1993
Return Test Products to RFP	August-September, 1993

Figure 7.9-1 Tentative Schedule for the Plutonium in Soils Treatability Studies Work Plan: TRU Clean Process

8.0 REFERENCES

ACGIH, 1990. Threshold Limit Values and Biological Exposure Indices, Cincinnati, OH.

AWC, 1987. "The Removal of Plutonium Contaminants from Rocky Flats Plant Soil (1987)," AWC Inc., Las Vegas, NV

DOE, 1991. Final Treatability Studies Plan, U.S. Department of Energy, Rocky Flats Plant, June 3, 1991.

DOE, 1982. Navratil, J.D. and Kochen, R.L., "Decontamination of Soil Containing Plutonium and Americium," Rocky Flats Plant, RFP-3139.

EG&G, 1990. General Radiochemistry and Routine Analytical Services Protocol Scope of Work, EG&G Rocky Flats, 1990.

EG&G, 1991. Site-Wide Quality Assurance Project Plan, Rocky Flats Plant, May 5, 1991.

EPA, 1986. Test Methods for Evaluating Solid Waste Physical/Chemical Methods, U.S. Environmental Protection Agency, 1986.

EPA, 1987. Data Quality Objectives for Remedial Response Activities, U.S. Environmental Protection Agency, EPA 540/G-87/003A, March, 1987.

Tyler, 1989. Testing Sieves and Their Uses, Tyler, W.S., Handbook 53, 1989 Edition.

USDA, 1980. Soil Survey of Golden Area, Colorado, U.S. Department of Agriculture, Soil Conservation Service, 1980.

Weast, 1982. CRC Handbook of Chemistry and Physics, Weast, R.C., et al, 63rd Edition, 1982.

ATTACHMENT 2

TABLE OF CONTENTS
STANDARD OPERATING PROCEDURES
AWC

SOP No.	TITLE	REV.	CHG.	EFFECTIVE DATE
SOP NTS 101	Operation of Gravimetric Separator	1		7/01/91
SOP NTS 102	Operation of the Centrifugal Separator	1		7/01/91
SOP NTS 104	Operation of the Multi-Gravity Separator	1		7/01/91
SOP NTS 201	Operation of Trommel	1		7/01/91
SOP NTS 202	Operation of Feed Auger	1		7/01/91
SOP NTS 203	Operation of the Attrition Scrubber	1		7/03/91
SOP NTS 301	Operation of the Spiral Classifier	1		7/01/91
SOP NTS 302	Operation of the Hydrocyclone Test Unit	1		7/01/91

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ANALYTICAL PROCEDURES
BULK DENSITY DETERMINATION

AP No. AWC 2
Revision 0
Page 1 of 22

Approvals:

Terry Wenstrand	5/2/91	Gregory Hodie	5/2/91
Project Manager	Date	Radiation Safety Officer	Date
Gregory Hodie	5/2/91	Gregory Hodie	5/2/91
QA Manager	Date	E,S&H Coordinator	Date

1.0 PURPOSE AND SCOPE

The purpose of this analytical procedure (AP) is to instruct laboratory personnel in the methods used for determining the bulk density of soil samples.

2.0 APPLICABILITY

This AP applies to laboratory personnel involved in soil analysis activities in which the bulk density must be determined.

3.0 DEFINITIONS

3.1 Bulk Density

Weight of dry soil per unit volume including the pore space.

3.2 Clod

A self-adhering piece of earth collected away from any digging shear face.

CAUTION: Impermeable rubber gloves must be worn during all procedures involving soils suspected to contain radionuclides.

4.0 RESPONSIBILITIES

The laboratory supervisor is responsible for assuring that all personnel assigned tasks related to this AP are familiar with this AP, that they have been trained in this AP, and that a controlled copy of this AP is available to them.

All personnel performing bulk density determination are responsible for following this AP.

5.0 PROCEDURE

Discussion

Density is defined as weight per unit volume and is expressed in units of g/cm^3. The bulk density of a soil is defined as the weight of dry soil per unit volume including the pore space. For mineral soils, bulk density generally ranges from 0.6 to 2.0 g/cm^3. With increasing organic matter content, soils generally exhibit a decrease in bulk density because organic matter has higher porosity and lower density than mineral particles of the same diameter.

The clod method is the primary method for determining bulk density. Where possible, three replicate clod samples are extracted from each horizon. The average bulk density of the replicates is assumed to be the bulk density of that particular horizon. Analysis of the clods is based on the method described in the USDA/SCS (1984), Kern and Lee (1989), and Kern et al. (in preparation).

Two alternate methods are also presented for soil horizons that fail to yield satisfactory clods. One method is volume replacement (VR), a method similar to one described by Flint and Childs (1984), which utilizes a known volume of small foam beads packed into a cylinder to replace a selected volume of soil excavated from a given horizon. Subtracting the initial from the final volume yields the estimated volume of sample collected. The other method is a volume filling (VF) method that is used if the clod or VR methods do not produce representative samples. The volume of this type of sample is based on the absolute volume of a 250-mL beaker, which is a constant 300 cm^3. The known volume samples are processed in a manner similar to the method described in Blake (1965). Whenever possible, use the clod method.

Interferences

Evaporating dishes used in the clod method must be thoroughly cleaned after each use, as their weights are pre-determined and used in the calculation of results. If soils are suspected to contain radionuclides impermeable gloves must be used when transferring the dishes from one location to another.

5.1 Sample Collection, Preservation, and Storage

Clod samples are fist-sized, structurally intact, bulk density samples taken from designated horizons. In the field, each clod is wrapped in a haimet and dipped briefly in a 1:5 Saran:acetone solution to help maintain the clod structure and reduce moisture loss from the clod during transport and storage. Each clod is labeled, covered by a small plastic bag, and placed in a divided box. Clods must be collected away from shear faces.

Known volume bulk density samples are taken from horizons where clods are unobtainable. There are two types of known volume samples, volume replacement and volume filling samples. These bulk density samples are packaged in small, pre-labeled plastic bags.

Samples are stored at 4 °C ± 2° C until analysis. If the analysis is for only radionuclides this temperate control can be ignored.

5.2 Equipment and Supplies

5.2.1 Apparatus

The following items are used in both the clod and volume methods:

1. Drying oven or convection oven, if available .

2. Desiccator and desiccant.

3. Brass sieve, squared-holed, 2 mm.

4. Forceps.

The following additional apparatus is needed for the clod method:

1. Fume hood.
2. Balance, capable of weighing to 0.1 g.
3. Beakers, 1000-mL and 2000-mL.
4. Ring stand or similar apparatus.
5. Test tube clip, adjustable, or similar apparatus.
6. Evaporating dishes, pre-numbered, tolerance to 450 °C.
7. Furnace gloves (optional).
8. Furnace tongs.
9. Muffle furnace.
10. One-gallon metal cans with airtight lids (paint cans).
11. Magnetic stirrer and stir bars, or long stir sticks.
12. Shower curtain rod or similar apparatus.
13. Stop watch.
14. Thermometer.
15. Balance calibration weights, 3-5 weights covering expected range.

16. Class "P" balance weight, 500 g.

The only item needed for the volume methods in addition to the four listed previously is:

- Balance, accurate to 0.01 g.

5.2.2 Reagents

NOTE: All reagents listed are for the clod method. The volume methods do not require any reagents.

1. Acetone, industrial grade.

2. Saran:acetone clod dipping mixture, approximately 1:5 by weight.

NOTE: The clod dipping solution must be mixed only under a properly operating vented fume hood. The operator must wear a laboratory coat while mixing the dipping solution.

The Saran powder is prepackaged in 540-g allotments. One packet mixed with approximately 3400 mL of acetone provides a nearly 1:5 by weight mixture resembling light syrup. If possible, use a magnetic stirrer and a stir bar to mix the ingredients in a beaker until well blended, then transfer mixture to paint container for storage. One-gallon paint containers are used for mixing and storing the solution. Slowly add the contents of each packet to the acetone while stirring. Continue stirring to ensure thorough dissolution of the powder. The solution normally is well mixed when it has the color and consistency of amber syrup and there is no gummy Saran residue on the edges or bottom of the container. If the solution is a milky color, additional mixing is

required. The powder has a tendency to precipitate and clump around a container's lower rim when mixed with acetone. To prevent this, use a long stir stick and thoroughly scrape the inner rims.

After mixing the solution, the container should be capped as tightly as possible to prevent leakage of the solution or evaporation of the acetone.

3. Water – Tap grade or better water may be used. Allow the water to reach room temperature before using.

5.2.3 Consumable Materials

Consumable items required for the clod method are:

1. Paper bags, pre-labeled.

2. Plastic bags, large.

3. Clod bulk density raw data form (Attachment 10.1).

Consumable items required for the known volume methods are:

1. Paper bags, pre-labeled.

2. Known volume bulk density raw data forms (Attachment 10.2).

3. Known volume log book (known volume method).

4. Volume data from sample receipt log book (known volume method).

5.3 Calibration and Standardization

Standardization of the balance is detailed in AP No. 1, General laboratory Procedures.

Check thermometers monthly against an NBS Traceable Standard thermometer to ensure that they are measuring temperature accurately. If a thermometer varies by more than 2° C from the NBS Traceable Standard, it will be removed from service.

5.4 Clod Method

NOTE: All raw data obtained from the clod analysis are recorded on the clod bulk density raw data form (see Attachment 10.1). For each clod to be analyzed, record the set ID, sample code, and the replicate number on the form. Examine the label attached to the clod and record the number of field dips performed by the sampling crew under "FIELD_DP" on the form. Weigh the clod, without removing the label, to the nearest 0.1 g and record this weight under "FIELD_WT/MOIST".

1. Suspend the clod in a convection oven and dry for at least 12 hours or overnight at 105 °C for mineral samples, or 60 °C for organic samples. After drying, transfer the clod into a desiccator and allow it to cool for 30 minutes. Weigh the sample to the nearest 0.1 g and record this weight under "FIELD_WT/DRY".

2. Hang the clod on a suspended rod within an operating vented fume hood. Dip the clod into a container of 1:5 by weight Saran:acetone mixture (see Section 5.2.2, Step 2) for three seconds and then allow the coating to dry for approximately 15 minutes. Do not allow the mixture to impregnate that portion of the haimet hanging above the top of a clod.

NOTE: Alternately, the clod may be sprayed with water just prior to dipping in the Saran:acetone solution. The water reduces the penetration of Saran into the clod and produces a frost appearance, indicating the solution has coated the outside of the clod. Whichever alternative is chosen, it is important that the same technique be used on all samples.

3. Apply additional dips as necessary until the clod does not produce excessive bubbles and a coating of Saran is obtained that appears to be impervious to water. Usually three to six dips is sufficient for most clods. Record the total number of dips made in the laboratory under "LAB_DP", then reweigh the clod to the nearest 0.1 g and record this weight under "LAB_WT". Also perform and record the dry-weight quality control check (Section 6.0).

4. Add approximately 1600 mL of deionized water to a 2000-mL beaker. Place the beaker of water on a balance and tare. Record the temperature of the water under "TEMP" so that the density of the water can be calculated. Also perform and record the submerged weight quality control check (Section 6.0).

5. Suspend the clod over the beaker by attaching the top of the hairnet to the test tube clip, then lower the clod gently into the water until the top of the clod is entirely submerged. Record the weight displayed on the balance to the nearest 0.1 g under "CLOD_H2O". If the clod floats, forcibly submerge it by pushing down with forceps and record the weight. Note on the raw data form, with a "Y" under "FLOAT", that the clod floated. Retare the balance before proceeding with other clods.

   NOTE: When submerging, ensure that the clod is not touching the edge of the beaker and that the clod label hangs freely. Occasionally, air bubbles may rise from the clod and the weight reading on the balance does not stabilize but steadily decreases. This occasionally occurs in clods sampled from relatively porous surface horizons and simply indicates that all of the primary macropores along the exterior of the clod were not thoroughly water-sealed. By reading the weight immediately after submersion, error from this source of instability is minimized. Note the bubbling on the raw data form under "COMMENTS". Also note any other problem, such as a broken clod, under "COMMENTS".

6. After submersion, place the clod in a pre-numbered evaporating dish and record the dish number on the raw data form under "CRUC_NO". Remove as much of the clod label and haimet as possible and place in a large plastic bag with others from the same run.

7. Place the evaporating dish plus clod in a muffle furnace equilibrated at 400 °C. The muffle furnace must be within an operating vented fume hood. Allow the Saran coating to be burned off the clod surface for two hours. Allow the furnace to cool, then carefully remove the evaporating dish from the muffle furnace. After the clod has cooled thoroughly, place it in a pre-labeled paper bag. If the soil is suspected to contain radionuclides place the paper bag containing the clod into a plastic bag.

8. Disaggregate the clod through a 2-mm sieve. Weigh any rock fragments retained on the sieve to the nearest 0.01 g and record this weight under "R_FRAG". Place the rock fragments into the paper bag and archive all such bags.

   CAUTION: If the soil is suspected to contain radionuclides perform this operation in an approved fume hood.

   Alternatively, the clods may be soaked in water and dispersing agent to aid in disaggregation followed by drying of coarse fragments at 105°C.

5.5 Known Volume Method

NOTE: Known volume bulk density samples are shipped from the field in small, plastic bags. Upon passing the sample receipt verification, these samples can be placed out to air dry in the drying area (see Section 5.5.1). The drying facilitates the transfer of the entire sample from its plastic bag into a pre-labeled paper bag. If sample is suspected to contain radionuclides, this drying area must be in an approved fume hood. Do not remove any material from the bag, even if the material is thought to be morphologically different from the type of horizon sampled.

NOTE: Record the set ID and sample code of the VR or VF sample on the known volume bulk density raw data form (see Attachment 10.2).

5.5.1 Laboratory Measurements

When clods cannot be obtained, the volume-replacement method is used. A level step is prepared in the soil pit at least large enough to dig two circular holes approximately 10 cm in diameter and 10 cm deep, or to the boundary of the next lowest horizon. Larger diameter holes must be used for thin horizons. When a level step cannot be obtained a metal frame is used to provide a reference point for calibrating the surface roughness (see Section 5.5.1.1). All material (including roots) excavated from the small holes is placed into separate sample bags, paying careful attention to retain all of the sample. Label each sample bag.

The volume of each hole is determined by lining the hole with cheesecloth by carefully pushing the material into all the irregularities of the hole. Fill a plastic graduated cylinder with beads. Record notes in a field logbook indicating which sample is being measured (i.e., 1/2 for the first of two). Read the initial volume of the beads in the cylinder by placing the circular block into the cylinder and tapping the side twenty times (or more until large voids not visible) with a trowel handle. Record the initial volume in a field logbook as V_i. Fill the fabric lined hole with beads, tamping and then levelling them. Use a straight-edge to help judge when the hole is full. Record the final volume of beads in the cylinder as V_f. Remove the beads and repeat for the second hole.

Seal the plastic bags as instructed for routine samples and place all the volume-replacement samples for one horizon in a canvas bag that is labelled with "Volume-Replacement," date sampled, crew ID, site ID, and set ID.

5.5.2 Volume-Replacement Sampling with Frame

In cases where a level step cannot be obtained due to rock fragments or abundant roots the volume-replacement method must be used with a metal

frame. When the metal frame is used with this method, only one replicate is required, instead of the usual two, because it is more time consuming. The volume measurements are recorded in the field logbook.

Two volume measurements are to be made when using this method. The first is to calibrate the volume from the top of the frame to the soil surface. The second measurement is made after a hole has been excavated within the area enclosed by the frame. The prep lab will subtract the second measurement from the first to obtain the volume of soil excavated.

Prepare a level step, as best possible, then position the metal frame and secure it with nails. Line the inside of the frame with cheesecloth and tamp it into all corners. Place beads into the graduated cylinder and tamp it as described above. Fill the frame with beads, tamping them gently while filling. Record the amount of beads used in the field logbook. It may take more than one cylinder of beads to fill the frame, in which case, record in the "V_i" space the total volume in the first cylinder and write the volume in the second cylinder before pouring beads into the frame. For "V_f" write the volume in the last cylinder after filling the frame to the rim. For example, if 1,900 cm^2 of beads were placed in the frame and then the cylinder was filled back up to 1,800 cm^2, the entry for "V_i" calibration would be "1,900 + 1,800." If after filling the frame to the rim, striking it off with a straight-edge and returning the excess beads to the cylinder, the volume in the cylinder was 1,500, then the entry for "V_f" is 1,500.

Remove the beads from the frame used for calibration and place in a plastic bag. Excavate a sample, as described above, from a hole within the frame and label it. Line the hole and frame with cheesecloth. Pour all the beads used for calibration into the cheesecloth and write in the "V_i" section for final volume "calibration beads +." Fill a graduated cylinder with beads and record the volume in "V_i" for initial volume. Pour beads from the cylinder until the frame is filled. Strike off the top surface of the frame with a straight-edge and return the excess

beads to the cylinder. Record the final volume in the cylinder in the space for "V_f."

1. Weigh the paper bag plus sample to the nearest 0.01 g and record this weight under "AIR_WT".

2. Place the bag in a drying oven equilibrated at 105 °C ± 5° C for mineral soils and 60 °C ± 5° C for organic soils. Allow the sample to oven dry overnight.

3. The following morning, remove the sample from the oven and allow to cool for 30 minutes. Weigh the sample to the nearest 0.01 g and record this weight under "OD_WT".

4. Sieve the contents of the bag through a 2-mm sieve to remove any rock fragments. Weigh the rock fragments to the nearest 0.01 g and record this weight under "R_FRAG". Place the rock fragments in the paper bag and archive all such bags.

5. Transcribe the computed volume (V_i minus V_f) for a VR sample from the sample receipt log book to the "VOL" column on the raw data form. For a VF sample, 300 cm^3 can be entered under "VOL"

5.5.3 Sample Drying

1. Label a bulk sample processing raw data form (see attachment 3) for each sample to be air dried.

2. Place two fresh sheets of kraft paper, approximately 1m x 1m in area, on the mesh partition of the drying table. With gloved hands, slowly spread the sample on top of the sheets of paper, taking care not to lose any soil off the paper or contaminate any adjacent samples. If a sample is

suspected to contain radionuclides, it must be dried in an approved fume hood.

NOTE: In some cases, samples may be received from the field in more than one sample container (bag). Combine the contents of all containers comprising a single sample.

3. Disaggregate any large clumps that impede the spreading of the sample over the entire area of the paper. Place an additional sheet of kraft paper loosely over the sample.

4. Identify the sample by attaching the sample's processing raw data form to a hook attached to the drying table or other mechanism. The original canvas and plastic sampling bags should be kept on the floor beneath the sample as a second check on sample identity.

5. Daily, stir the soil sample with gloved hands to facilitate drying.

6. For the first few days that a wet sample is spread, the bottom sheet of paper may need to be changed daily in order to alleviate excessive moisture accumulation. Any observations of fungal or algal growth should be noted on the bulk sample processing raw data form.

NOTE: Soils high in clay may harden nearly irreversibly if allowed to dry without a preliminary disaggregation of medium and coarse aggregates. An effort should be made during the air-drying procedure to disaggregate these pedons by physical manipulation with gloved hands while still somewhat moist or friable, before reaching an air-dry state.

7. Allow the sample to air dry until it is believed to be at or below the specified moisture content. This process generally takes about two days, although drying time may vary from one day to a week or more.

5.5.4 Volume-Filling Bulk Density Sampling

When the previous two bulk density methods do not work, the volume-filling method is used. This method is reserved for material such as loose sand, which cannot maintain an excavation, or irregular surface horizons. Samplers fill plastic beakers cylinders of known volume as best they can to recreate the same packing as naturally found in the soil. Fill the container completely and strike off with a straight-edge. The material is carefully transferred to a plastic bag and labeled. Two samples will be taken each time this method is used.

Seal the plastic bags as instructed for routine samples and place all the volume-filling samples for one horizon in a canvas bag that is labelled with "Volume-Filling," date sampled, crew ID, site ID, and set ID.

5.6 Calculations

5.6.1 Clod Method

It is assumed that the weight of each two-second Saran coating applied in the field is equal to the weight of each three-second coating applied in the laboratory, as some of the dipping solution normally is absorbed into the clod when it is applied in the field.

It is assumed that the specific gravity of air-dry Saran is 1.30 g/cm^3 and that the coating loses 15 percent of its weight upon oven-drying. It is assumed that the particle density of the rock fragments is 2.47 g/cm^3.

It is assumed that the average gross weight of the haimet and clod label is 1.88 g and the net weight of the submerged portion of the haimet is 0.20 g.

The following calculations may be performed by computer after the raw bulk density data are entered. If so, it is advantageous to manually check a few samples using these calculations to understand the procedure and to test the accuracy of the computer program. A list of temperature-corrected water density values is provided in Table 5-1.

Table 5-1. Density of Pure Water[a]

Temp °C	Water Density
13.0	0.9992
14.0	0.9991
15.0	0.9990
16.0	0.9988
17.0	0.9987
18.0	0.9986
19.0	0.9984
20.0	0.9982
21.0	0.9980
22.0	0.9978
23.0	0.9976
24.0	0.9973
25.0	0.9971
26.0	0.9968
27.0	0.9965
28.0	0.9963
29.0	0.9960
30.0	0.9957
31.0	0.9954
32.0	0.9951
33.0	0.9947
34.0	0.9944
35.0	0.9941

[a] Adapted from List (1984).

Use the following calculations to derive the weights and volume of the air-dry and oven-dry Saran coatings:

$$\text{SARAN_WT (g)} = \frac{[(\text{FIELD_DP} + \text{LAB_DP}) \times (\text{LAB_WT} - \text{FIELD_WT/DRY})]}{\text{LAB_DP}}$$

$$\text{OD SARAN_WT (g)} = \text{SARAN_WT} \times 0.85$$

$$\text{SARAN VOL (cm}^3\text{)} = \frac{\text{SARAN_WT}}{1.30}$$

Use the following calculations to derive the volumes of rock fragments, water displacement, and soil/pore fraction:

$$\text{R_FRAG VOL (cm}^3\text{)} = \frac{\text{R_FRAG}}{2.47}$$

$$\text{WATER VOL (cm}^3\text{)} = \frac{\text{CLOD_H2O}}{\text{DENSITY OF WATER}}$$

$$\text{SOIL/PORES VOL (cm}^3\text{)} = \text{WATER VOL} \cdot _ \cdot (\text{R_FRAG VOL} + \text{SARAN VOL})$$

Finally, use the following calculation to derive the oven-dry bulk density (BD_CLD) for the individual clod:

$$\text{BD_CLD (g/cm}^3\text{)} = \frac{\text{LAB_WT} \cdot _ \cdot (\text{R_FRAG} + \text{OD SARAN_WT} + 1.88)}{\text{SOIL/PORES VOL} \cdot _ \cdot 0.20}$$

5.6.2 Known Volume Method

The particle density for rock fragments in a VR bulk density sample is derived individually by a submersion technique similar to that used in the clod analysis. It is assumed that the particle density for rock fragments in a VF bulk density sample is 2.65, unless specific particle density measurements are made of different classes of rock fragment types.

The following calculations for known volume bulk density (BD_KV) may be performed by computer. It is advantageous to manually check a few samples using these calculations to understand the procedure and to test the computer program.

$$\text{FINES_WT (g)} = \text{OD_WT} \cdot _ \cdot \text{R_FRAG}$$

$$\text{BD_KV (g/cm}^3) = \frac{\text{FINES_WT}}{\text{VOL} \cdot _ \cdot (\text{R_FRAG} + \text{particle density})}$$

6.0 QUALITY CONTROLS

NOTE: This applies only to the clod method.

Record the dry weight and suspended submerged weight of a 500-g balance weight immediately before and after a run of samples. These weights are used to verify proper balance operation and should be charted on a daily basis whenever bulk density determinations are made.

7.0 ENVIRONMENT, SAFETY AND HEALTH

7.1 Clod Method

Laboratory personnel should use caution when working around the muffle furnace because temperatures of up to 450 °C are common. The furnace should be activated only in an operable fume hood or other approved ventilation area. Heat resistant or furnace gloves may be needed when placing samples in the furnace. The furnace must be adequately vented and protected from incidental human contact and no combustible/flammable materials should be placed in or near the furnace.

Extra precaution must be taken in the use of the Saran powder and acetone solution (see Section 5.3.2, Step 2). When mixed, the resulting solution has a tendency to volatilize hydrogen chloride gas which can cause deleterious health effects. The solution should be used only in an operating vented fume hood. Half mask with appropriate cartridge

respiratory protection, gloves, and laboratory coat should be worn as specified in the Laboratory Run Sheet.

In the field, exposure to Saran solution through inhalation and skin contact must be minimized. Gloves and respiratory protection are recommended and will be specified in the Laboratory Run Sheet. Use solution only in a well-ventilated area which is downwind of the sampling site.

7.2 Volume Methods

The safety concerns cited above do not apply to the VR or VF methods. Protective clothing (laboratory coat and gloves), safety glasses, and dust masks should be worn.

8.0 RECORDS

The following documents will be maintained as Quality Records.

- Clod bulk density raw data
- Known volume bulk density data
- Bulk sample raw data
- Field logbook

9.0 REFERENCES

American Society for Testing and Materials. 1984. *Annual Book of ASTM Standards, Vol. 11.01, Standard Specification for Reagent Water,* D-1193-77 (reapproved 1983). ASTM, Philadelphia, Pennsylvania.

Blake, G. R. 1965. In Black, C. A. (ed.) *Methods of Soil Analysis, Part 1.* American Society of Agronomy, Madison, Wisconsin.

Flint, A. L., and S. Childs. 1984. Development and Calibration of an Irregular Hole Bulk Density Sampler. *Soil Science Society of America Journal* 48 (2):374-378. Soil Science Society of America, Madison, Wisconsin.

Kern, J. S., and J. J. Lee. 1989. Evaluation of Two Alternative Bulk-Density Methods. *Agronomy Abstracts*. American Society of Agronomy, Madison, Wisconsin.

Kern, J. S., M. L. Papp, J. J. Lee and L. J. Blume. In preparation. Appendix A In Kern, J. S. and J. J. Lee, *Direct/Delayed Response Project: Field Operations and the Mid-Appalachian Region of the United States*, U.S. Environmental Protection Agency, Office of Research and Development, Washington, D.C.

List, R. J. 1984. *Smithsonian Meteorological Tables*, 6th Edition. Smithsonian Institute Press, Washington, D.C.

USDA/SCS. 1984. *Soil Survey Laboratory Methods and Procedures for Collecting Soil Samples. Soil Survey Investigations Report No. 1.* U.S. Government Printing Office, Washington, D.C.

10.0 ATTACHMENTS

Attachment 10.1 "Clod bulk density raw data" form

Attachment 10.2 "Known volume bulk density data" form

Attachment 10.3 "Bulk sample raw data" form

CLAY BULK DENSITY RAW DATA

page ___/.

ALL WEIGHTS RECORDED TO 0.01 GRAMS

SAM_CODE	REP	FLD_BY	FLD_WT mist/dry	LAB_BY	LAB_WT	CLAY_STO	TEMP C		CRUC NO.	S_FRAC		FLOAT	PROB T/T	ID	COMMENTS

AWC

KNOWN VOLUME BULK DENSITY DATA

	AIR WT.	OD WT.		VOL. (cc)	DATE	ID	COMMENTS	SAMPLER (INITIALS)	DATE

Supervisor _____ Date _____

AWC

BULK SAMPLE RAW DATA

SAMPLE ID:_____ DATE SAMPLED: ___/___/___

SITE ID:_____ DATE RECEIVED: ___/___/___

SET ID:_____ PROCESS START: ___/___/___

BATCH ID:_____ PROCESS COMPLETE: ___/___/___

SOIL TYPE: M / O FIELD pH: _____

Initials: _____ Initials: _____

AIR SAMPLE DRYING:

Date	% Moisture	Initials
___/___	_____	_____
___/___	_____	_____
___/___	_____	_____

TOTAL BULK WT:

Date: ___/___/___ _____.____ g Initials: _____

ROCK FRAGMENT WT:

2 TO 4.75 mm: _____.____ g 4.75 to 20 mm: _____.____ g

Date: ___/___/___ Initials: _____

ENTERED IN COMPUTER: Date: ___/___/___ Initials: _____

COMMENTS:

_____ _____
Supervisor Date

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ANALYTICAL PROCEDURES
PARTICLE SIZE ANALYSIS

AP No. AWC 3
Revision 0
Page 1 of 14

Approvals:

Terry Wenstrand	5/2/91	Gregory Radie	5/2/91
Project Manager	Date	Radiation Safety Officer	Date
Gregory Radie	5/2/91	Gregory Radie	5/2/91
QA Manager	Date	E,S&H Coordinator	Date

1.0 PURPOSE AND SCOPE

The purpose of this analytical procedure (AP) is to instruct laboratory personnel in the methods used in particle size analysis.

Particle size analysis is determined on the less than 1.25-cm fraction for mineral and activity horizons. The sieve/pipet/gravimetric method described in Methods of Soil Analysis (ASA/SSSA, 1986) is used with adaptions for activity analysis.

The soil sample is first dry sieved and then each of the seven fractions weighed and assayed for radioactivity. The soil fractions are returned to their respective screens and wet sieved followed by drying, weighing, and assaying for radioactivity. The soil fraction collected in the pan is suspended in water; aliquots taken from the suspension under specific conditions are dried, weighed, and assayed for radioactivity.

2.0 APPLICABILITY

This AP applies to laboratory personnel involved in particle size analysis of soil samples.

3.0 DEFINITIONS

SRCC - Standard Radiological Counting Can

4.0 RESPONSIBILITIES

The laboratory supervisor is responsible for assuring that all personnel assigned tasks related to this AP are familiar with this AP, that they have been trained in this AP, and that a controlled copy of this AP is available to them.

All personnel performing particle size analysis of soil samples are responsible for following this AP.

5.0 PROCEDURE

Interferences

While the soil in suspension is settling, the graduated cylinders containing the suspension cannot be disturbed, nor can the temperature vary. Foam insulation, a constant temperature water bath, or temperature-controlled room may be used to maintain constant temperature. When handling weighing bottles, use forceps, finger cots, cotton gloves, or vinyl gloves to avoid adding weight from moisture and from body salts and oils.

5.1 Particle Size Sample Collection

The subsamples for particle size analysis are taken from the bulk soil sample after it has been air dried and homogenized.

5.2 Equipment and Supplies

5.2.1 Apparatus

1. Sedimentation cylinders (1-L graduated cylinders, optional).

2. Stirrer, hand–Fasten a circular piece of perforated plastic to one end of a brass rod.

3. Shaw pipet rack or equivalent (preferred).

4. Pipets, 25-mL, automatic (Lowry with overflow bulb or equivalent).

5. Sieve shaker, 1.25-cm vertical and lateral movement, and 500 oscillations per minute, or equivalent. Unit must accommodate a nest of sieves.

6. Weighing pans, 20-cm diameter by 5 cm deep, or equivalent, (tare to ±0.1 mg), capable of withstanding intermittent heating to 110 °C and cooling to room temperature.

7. Balance, top-loading, capable of weighing to 0.01 g.

8. Balance, analytical, capable of weighing to 0.0001 g.

9. Set of sieves, square-mesh, woven phosphor-bronze or stainless steel wire cloth; U.S. Series and Tyler Screen Scale equivalent designations as follows:

Nominal Opening (mm)	U.S. No.
2.0	10
0.85	20
0.30	50
0.15	100
0.075	200
0.045	325

10. Receiving pan, used with sieves.

11. Hot plate (block digester, optional).

12. Thermometer, range 10 to 50 °C.

13. Evaporating dishes, or equivalent, 125- or 250-mL.

14. Desiccator and desiccant.

15. Planchets.

16. Standard Radioactivity Counting Cans (SRCC).

17. Drying oven or Convection oven.

18. Clamp and ring stand or equivalent.

19. Stoppers (optional).

20. Stop watch or other timer.

5.2.2 Reagents and Consumable Materials

1. Sodium carbonate (Na_2CO_3).

2. Sodium hexametaphosphate [$(NaPO_3)_6$], dispersing agent–Dissolve 35.7 grams of $(NaPO_3)_6$ and 7.94 grams of Na_2CO_3 in deionized (DI) water and dilute to 1 L.

3. Water–Commercial DI water is suitable for these procedures.

4. Kimwipes or equivalent.

5. Forms–Form 2 particle size parameters raw data, Form 3 particle size parameters calculated data, Form QC-2, and Form QC-3 (Attachments 10.1 through 4, respectively).

5.3 Calibration and Standardization

Check thermometers monthly against an NBS traceable standard thermometer to ensure that they are measuring temperature accurately.

If a thermometer varies by more than 2 °C from the NBS standard it will be removed from service. Temperatures of the suspensions used in these procedures should vary no more than ±1 °C.

Standardization the balance is described in AP No. 1, General Laboratory Procedures.

5.4 Dry Sieving Method

CAUTION: Wear impermeable rubber gloves when handling any sample suspected to contain radionuclides.

1. Set up sieve shaker in hood with #10, #20, #50, #100, #200, #325 sieves and receiving pan.

2. Weigh approximately 1000 grams of sample and record weight on dry sieve data sheet (Attachment 10.1).

3. Place sample in drying oven at ± 10°C for 1 hour beyond apparent dryness.

4. Cool sample to room temperature.

5. Remove sample from oven and weigh, recording weight on dry sieve data sheet.

6. Place sample in top sieve (#10), place cover over #10 sieve and secure in shaker.

7. Start sieve shaker and shake for 10 minutes.

8. Remove #10 screen and transfer soil fraction into a tared SRCC. Weigh SRCC, subtracting tare weight and record on dry sieve data sheet.

9. Repeat step 8 for each sieve and receiving pan.

   NOTE: If the sieve fraction will not fit into one SRCC, place remaining soil fraction into second SRCC.

10. Count each SRCC containing a sample for radioactivity and record results on dry sieve data sheet using the appropriate calibrated radiological detection system.

11. Complete mass and activity balance on dry sieve data sheet.

5.5 Wet Sieving Method

1. Replace soil fractions removed from dry sieving procedure back into their respective pans starting with the #325 sieve.

   NOTE: Do not replace material from receiving pan.

2. After all material has been replaced into their respective pans, wash #10 sieve with a squirt bottle while agitating soil with a rubber policeman. Wash until the water coming through the sieve is nearly clear. Large samples can be more effectively processed by slurrying in a pan and then pouring into the sieve. Repeat until the effluent is nearly clear.

3. Following washing of #10 sieve, transfer sieve contents to a tared drying pan and dry in oven at 110°C ± 5 °C for 1 hour beyond apparent dryness.

4. Repeat step 3 for the remaining sieve fractions working from the #20 sieve to the #325 sieve.

5. Each dried sieve fraction is transferred to a tared SRCC and the sample weight recorded on the wet sieve data form.

   NOTE: If the sieve fraction will not fit into one SRCC place remaining soil fraction into second SRCC.

6. The material collected in the pan is dried in a drying oven until a thin film of water remains over sample. Do not allow any dry areas to form in slurry.

   Combine dried material from receiving pan with material retained from receiving pan in dry sieving and mix to obtain homogeneous sample. Transfer material to tared tuna fish can for weight determination and radioactivity assay. Record weight and radioactive assay results on wet sieve data sheet.

7. Count each SRCC for radioactivity and record results on wet sieve data sheet.

8. Complete mass and activity balance on wet sieve data sheet.

5.7 Pipetting Method

CAUTION: Wear impermeable rubber gloves when handling samples suspected to contain radionuclides.

All pipetting must be performed in a location free from drafts and temperature fluctuations. A temperature-controlled room, constant-temperature water bath, or foam insulation may be used.

1. Add 10 ml of 0.5 g/L Na - hexametaphosphate (HMP) solution to a 1 liter sedimentation cylinder. Dilute to 1000 ml with DI water.

2. Weigh 100 grams of soil from the receiving pan (combined wet & dry sieve receiving pan sample) and add to the sedimentation cylinder. Shake cylinder or stir to release trapped gasses from the soil. The 100 grams of soil should not include the moisture content of the soils. The moisture content can be determined by drying a small subsample of pan fraction.

3. Allow 12 to 24 hours for the temperature of suspension to equilibrate to room temperature. If water is stored at room temperature, one to two hours is adequate.

4. Stir the material in the sedimentation cylinder for 6 minutes with the motor-driven stirrer. Stir 8 minutes if suspension has been standing for more than 16 hours.

   NOTE: If stoppers of adequate size are available, it is preferable to stopper the cylinder, invert, and swirl. Care must be taken to ensure that the stopper is held tightly in the cylinder. Repeat this procedure at least six times. Inspect the bottom and sides of the cylinder to ensure that fine particles are not adhering to the glass walls of the cylinder.

5. Remove the stirrer and either (1) cover the cylinder with a length of polyurethane foam pipe-insulation, (2) immerse the cylinder in a constant-temperature water bath, or (3) place the cylinder in a temperature-controlled room.

6. Stir the suspension for 30 seconds with a hand stirrer; use an up-and-down motion. Record the time when the stirring is complete. Do not move, stir, or otherwise disturb the cylinder from this point until all pipetting has been completed.

7. Take the temperature of the solution in the cylinder by gently lowering a thermometer 5 cm into the suspension. Support the thermometer with a clamp to reduce disturbance to the suspension.

8. Use the temperature and Table 5-1 to determine the settling time required for the 20 micron fraction, e.g., at 22 °C allow 4 minutes, 35 seconds.

Table 5-1. Sedimentation Times for Fine Silt and Clay Particles Settling Through Water to a Depth of Ten Centimeters

Temperature	Settling Time with Indicated Particle Diameter[a]		
	2 microns	5 microns	20 microns
°C	hr:min	min:sec	min:sec
20	8:00	68:18	4:48
21	7:49	66.42	4:41
22	7:38	65:06	4:35
23	7:27	63:36	4:28
24	7:17	62:06	4:22
25	7:07	60:42	4:16
26	6:57	59:18	4:10
27	6:48	58:00	4:04
28	6:39	56:48	4:00
29	6:31	55:36	3:55
30	6:22	54:24	3:49

[a]Values calculated from Stokes' equations, assuming a particle density of 2.65 g/cm^3. This figure for particle density is arbitrary and has been chosen to satisfy simultaneously the two definitions of the clay fraction, i.e., particles that have an effective diameter of 0.002 mm or less and particles that have a settling velocity of 10 cm in 8 hours at 20 °C.

9. Approximately 60 seconds before the sedimentation time has elapsed, slowly lower the Lowry automatic pipet 10 cm into the suspension. (A 25-mL volumetric pipet premarked for a 10-cm depth and clamped firmly in place on a stand may be used.)

10. At the appropriate time, slowly (allow about 12 seconds) fill the pipet. Carefully remove it from the suspension.

11. Wipe clean the outside of the pipet and empty the contents into a glass container, such as a 90-mL widemouth bottle. Rinse the pipet into the bottle once with DI water (add the rinse water to the contents of the bottle). Transfer contents of glass container into tared planchet in 10 ml increments. Evaporate the water slowly with heat lamp or on hot plate (no splattering or boiling). Rinse empty glass container with DI water and transfer rinsed solutions to planchet.

12. Cool planchet in desiccator and weigh to nearest 0.1 mg and record net weight on wet sieve data sheet.

13. Repeat steps 7 through 12 for the 5 µm and 2 µm clay fraction. The <2-µm fraction may be pipetted at a time between 5 and 8 hours depending on the temperature and the table used. The use of Table 5-1 and a depth of 10 cm is strongly recommended and is the easiest method.

6.0 QUALITY CONTROLS

Reagent Blanks–A 25-mL aliquot of the diluted dispersion solution (10.0 mL of the hexametaphosphate dispersion solution diluted to 1 L), dried and weighed, is used as the clay reagent blank. One blank should be analyzed with each batch of ten samples or weekly if fewer than ten samples are run in that week.

Replicates--One sample from each batch of ten samples should be analyzed in duplicate.

7.0 ENVIRONMENT, SAFETY AND HEALTH

Forceps, tongs, or heat-resistant gloves should be used to handle weighing bottles after removal from the oven. Wear protective clothing and/or safety glasses when handling reagents or materials known to contain radionuclides.

8.0 RECORDS

The following documents will be maintained as Quality Records.

- Field logbook
- Dry Sieve Data Sheet form
- Wet Sieve Data Sheet form
- Pipette Method Data Sheet form

9.0 REFERENCES

American Society for Testing and Materials. 1984. *Annual Book of ASTM Standard Specification for Reagent Water*, D-1193-77 (reapproved 1983). ASTM, Philadelphia, Pennsylvania.

U.S. Department of Agriculture/Soil Conservation Service. 1984. *Soil Survey Laboratory Methods and Procedures for Collecting Soil Samples.* Soil Survey Investigations Report No. 1, U.S. Government Printing Office, Washington, D.C.

10.0 ATTACHMENTS

Attachment 10.1 "Dry Sieve Data Sheet" form

Attachment 10.2 "Wet Sieve Data Sheet" form

Attachment 10.3 "Pipette Method Data Sheet" form

AWC

DRY SIEVE DATA SHEET

DATE_____ SAMPLE ID_____

TECHNICIAN _____ AS RECEIVED WEIGHT_____

DRIED WEIGHT _____

ISOTOPE_____

SIEVE SIZE	NET SIEVE SAMPLE WEIGHT (gm)	SIEVE ACTIVITY (pCi/gm)	SIEVE ACTIVITY (pCi)	PERCENT TOTAL WEIGHT (%)	PERCENT TOTAL ACTIVITY (%)
#10 (2000 μ)					
#20 (850 μ)					
#50 (300 μ)					
#100 (150 μ)					
#200 (75 μ)					
#325 (45 μ)					
PAN (<45μ)					
TOTAL					

_____ Supervisor

Date

AWC

WET SIEVE DATA SHEET

DATE_____ SAMPLE ID_____

TECHNICIAN _____ DRIED WEIGHT _____

ISOTOPE_____

SIEVE SIZE	NET SIEVE SAMPLE WEIGHT (gm)	SIEVE ACTIVITY (pCi/gm)	SIEVE ACTIVITY (pCi)	PERCENT TOTAL WEIGHT (%)	PERCENT TOTAL ACTIVITY (%)
#10 (2000 μ)					
#20 (850 μ)					
#50 (300 μ)					
#100 (150 μ)					
#200 (75 μ)					
#325 (45 μ)					
PAN (<45μ)					
TOTAL					

_____ Supervisor

Date

AWC

PIPETTE METHOD

SAMPLE ID _____

DRIED WEIGHT _____

ISOTOPE _____

DATE _____

TECHNICIAN _____

	NET SAMPLE WEIGHT (gm)	CORRECTED SAMPLE WEIGHT (gm)	PAN WEIGHT	SAMPLE ACTIVITY (pCi/gm)	SAMPLE ACTIVITY (pCi)	CORRECTED SAMPLE ACTIVITY (pCi)	PAN ACTIVITY	PERCENT TOTAL WEIGHT (%)	PERC TOT ACTI (%
45 µ									
20 µ									
5 µ									
2 µ									
TOTAL									

_____ Supervisor

_____ Date

CONTROLLED COPY No. 007

Approvals:

Terry Wenstrand	5/2/91	Gregory Gadie	5/2/91
Project Manager	Date	Radiation Safety Officer	Date
Gregory Gadie	5/2/91	Gregory Gadie	5/2/91
QA Manager	Date	ES&H Coordinator	Date

1.0 PURPOSE AND SCOPE

The purpose of this analytical procedure (AP) is to instruct laboratory personnel in the method used to determine the pH of soil samples.

The pH is defined as the negative logarithm of the activity of hydrogen ions (H^+). The H^+activity is a measure of the "effective" concentration of hydrogen ions in solution; it is always equal to or less than the true concentration of hydrogen ions in solution. Values range from pH 1 to pH 14, with pH 1 most acidic, pH 7 neutral (at 25 °C), and pH 14 most alkaline. Each pH unit represents a tenfold change in H^+activity (i.e., a pH 4 solution is 10 times more acidic than a pH 5 solution).

When the pH of a sample solution is measured, the hydrogen ions come into equilibrium with the ion exchange surface (glass) of a calibrated pH electrode, which creates an electrical potential. This voltage difference is measured by the pH meter in millivolts (mv), which is then converted and displayed as pH units.

2.0 APPLICABILITY

The following procedure was developed to standardize the measurement of pH in soils.

The applicable pH range for soil solutions is 3.0 to 11.0.

This AP applies to laboratory personnel tasked to determine the pH of soil samples.

3.0 DEFINITIONS

None

4.0 RESPONSIBILITIES

The laboratory supervisor is responsible for assuring that all personnel assigned the task of pH determination are familiar with this AP, that they have been trained on the AP, and that a controlled copy of the AP is available to them.

All personnel performing pH determination of soil samples are responsible for following this AP.

5.0 PROCEDURE

Discussion

Two suspensions of each soil sample are prepared, one in deionized (DI) water and one in 0.01 M calcium chloride ($CaCl_2$). The pH of each suspension is measured with a pH meter and a combination electrode. This method is modified from USDA/SCS (1984). The DI water pH is generally higher than the pH of the 0.01 M $CaCl_2$.

Interferences

Factors that normally affect the measurement of pH are (1) electrolyte content of the extractant; (2) soil-to-solution ratio; (3) temperature and CO_2 content of the extractant; (4) errors that occur with instrument calibration, standard preparation, and liquid junction potential; (5) organic and inorganic constituents; (6) length of time the soil and solution stand before they are measured; and (7) technique used in reading the sample suspension.

Soils high in salts, especially sodium, may interfere with the pH reading and the electrode response time. Clay particles may clog the liquid junction of the pH reference electrode, slowing the electrode response time; thoroughly rinse the electrode with DI water between sample readings to avoid this problem. Wiping the electrode dry with cloth, laboratory tissue, or similar materials or removing the electrode from solution when the meter is not on standby may cause electrode polarization.

The initial pH of a nonalkaline soil will usually be as much as 0.5 pH unit greater than the pH taken after the sample has set for 30 minutes or longer. The pH can vary as much as 1.0 pH unit between the supernatant and soil sediment. Always place the electrode junction at the same distance (approximately 3 mm) above the surface of the soil sediment to maintain uniformity in pH readings.

NOTE: The figures in the square brackets, [ ], represent the column of the appropriate form where the data are recorded.

5.1 Sample Collection, Preservation, and Storage

The subsamples for pH measurement are taken from the bulk soil sample after it has been air dried and homogenized. Samples should be stored at 4 °C ± 2 °C until ready for analysis. Preparation of the soil suspensions for pH measurement is described in Section 5.4.

5.2 Equipment and Supplies

5.2.1 Equipment Specifications

1. Digital pH/mV meter, capable of measuring pH to ±0.01 pH unit, potential to ±1 mV, and temperature to ±0.5 °C. The meter must also have automatic temperature compensation capability (Orion Model 611 or equivalent).

2. A combination pH electrode, made of high quality, low-sodium glass. At least two electrodes, one as a backup, should be available. Gel-type reference electrodes must *not* be used; an Orion Ross combination pH electrode or equivalent with a retractable sleeve is recommended.

3. Balance, accurate to ±0.001 g.

4. Balance calibration weights, 3-5 weights covering expected range.

5.2.2 Reagents

1. pH Calibration Buffers (pH 4.0, 7.0, and 10.0)–Commercially available pH calibration buffers (National Bureau of Standards [NBS]-traceable) at pH values of 4.0, 7.0, and 10.0 (two sets from different sources for calibration and quality control checks).

2. Buffer of pH 7.0 for quality control check standard (QCCS)–The QCCS can be purchased or it can be prepared from 0.05 M potassium hydrogen phthalate ($KHC_8H_4O_4$ or KHP). This buffer must be from a different container or lot than the NBS-traceable standards used for electrode calibration. Dry KHP for 2 hours at 110 °C, cool to room temperature in a desiccator. Weigh 10.21 g of KHP, dissolve it in DI water, and dilute the solution to 1.00 L. To preserve the KHP solution, add 1.0 mL of chloroform or one crystal (about 10 mm in diameter) of thymol per liter of the buffer solution. This solution has the following pH values at the temperatures given: 3.99 at 15 °C; 4.002 at 20 °C; 4.008 at 25 °C; and 4.015 at 30 °C.

3. Water–Commercial DI water

4. Calcium hydroxide–Dissolve 0.185 g $Ca(OH)_2$ in DI water and dilute to 1 L.

5. Hydrochloric acid (HCl)–Dilute 1 mL concentrated HCl to 1 L with DI water.

6. Stock calcium chloride solution ($CaCl_2$), 1.0 M–Dissolve 55.49 g of anhydrous $CaCl_2$ or 73.51 g of $CaCl_2 \cdot 2H_2O$ in DI water and dilute to 500 mL.

7. Calcium chloride, 0.01 M $CaCl_2$–Dilute 20 mL of stock 1.0 M $CaCl_2$ to 2.0 L with DI water. If the pH of this solution is not between 5 and 6.5, adjust the pH by addition of dilute $Ca(OH)_2$ or HCl, as needed.

8. Potassium chloride (3 M)–Dissolve 224 g KCl in DI water and dilute to 1 L.

9. Potassium chloride (0.1 M).

10. Sodium hydroxide (NaOH) pellets.

5.2.3 Consumable Materials

1. Beakers, plastic, or paper containers, 50-mL.

2. Glass stirring rods or disposable stirrers, one per sample.

3. Weighing pans, disposable.

4. Forms–Form 4 pH in water, Form 5 pH in 0.01 M calcium chloride, Form QC-4, and Form QC-5 (Attachments 10.1 through 4, respectively).

5.3 Calibration and Standardization

NOTE: For storage and readings, the electrode need only be immersed to cover the liquid junction of the reference electrode (typically about 3 mm). Rinse the electrode with DI water between each sample and each buffer to prevent solution carryover. Do not rub or blot electrode dry because this may produce a static electric charge and thereby polarize the electrode. Electrodes are stored in Im KCL.

5.3.1 Instrument Preparation

1. Plug in the instrument and verify that the control knob is on "STD BY."

   NOTE: If instrument is used frequently, leave on and in "STD BY" mode between uses.

2. Connect the combination electrode to the meter. Consult the pH electrode manual for the proper procedure.

3. Verify that the level of reference filling solution (3 M KCl) in the electrode is just below the fill hole and that the fill hole is uncovered during measurement (slide the plastic sleeve down).

5.3.2 Calibration with Buffers for pH less than 7.0

1. Check the meter temperature calibration daily with a beaker of room temperature DI water and an NBS-traceable thermometer. If the display differs from the NBS-traceable thermometer by more than 1.0 °C, complete adjustments as described in manufacturer's manual.

2. Pour fresh pH 7.00 and pH 4.00, buffer solutions into labeled 50-mL beakers (one "RINSE", one "CALIBRATION", and one "CHECK" beaker filled with each buffer). Rinse all beakers three times with buffer solutions and fill with the appropriate buffer solutions.

3. Rinse the electrode with DI water. Place the electrode into the pH 7.00 "RINSE" beaker and swirl for 40 seconds. Place the electrode into the "CALIBRATION" beaker, turn the knob to "pH", swirl for 30 to 60 seconds (or until the pH reading is stable), and read the value on the display. Consult the pH-temperature chart, Table 5-1. Use the "CALIBRATE" knob to adjust the pH reading on the meter to the theoretical pH of the buffer solution at the appropriate temperature.

4. Repeat Step 3 for pH 4.00 buffer using the "% SLOPE" knob to adjust the pH reading.

5. Repeat steps 3 and 4 until both the pH 7.00 and the pH 4.00 buffers agree with the theoretical pH of the buffer solution at the appropriate temperature.

6. Check the standardization using the buffer solutions in the "CHECK" beakers. If the values differ by more than ±0.03 units from the theoretical value, repeat the standardization process. When the meter standardization is acceptable, record the pH and temperature readings for each buffer solution in the pH logbook.

5.3.3 Calibration for Buffers for pH Measurements Greater than 7.0

Proceed as in Section 5.3.2 but substitute pH 7.0 buffer for pH 4.0 buffer and substitute pH 10.0 buffer for pH 7.0 buffer.

5.3.4 Maintenance

- Check the level of the 3 M KCl filling solution in the electrode. If it is more than 1 cm below the filling hole, replenish the solution from a bottle of electrode filling solution.

5.3.5 pH Meter Electronic Checkout

This procedure should be performed in accordance with the manufacturer's manual whenever a new pH meter is set up or when calibration problems occur.

Table 5-1. pH Values of Buffers at Various Temperatures

Temperature						
25 °C	0 °C	5 °C	10 °C	20 °C	30 °C	40 °C
1.68	1.67	1.67	1.67	1.67	1.68	1.69
3.78	3.86	3.84	3.82	3.79	3.77	3.75
4.01	4.00	4.00	4.00	4.00	4.02	4.03
6.86	6.98	6.95	6.92	6.87	6.85	6.84
7.00	7.11	7.08	7.06	7.01	6.98	6.97
7.41	7.53	7.50	7.47	7.43	7.40	7.38
9.18	9.46	9.40	9.33	9.23	9.14	9.07
10.01	10.32	10.25	10.18	10.06	9.97	9.89

5.3.6 Electrode Etching Procedure

NOTE 1: Use caution when using the NaOH pellets. Wear gloves, eye protection, and a lab coat.

NOTE 2: If the electrode response is sluggish or if the instrument cannot be standardized, the following procedure is recommended for cleaning the ceramic junction of the electrode and improving the electrode response time.

NOTE 3: Etch electrodes in groups of three when possible. Prepare a fresh NaOH solution for each group of electrodes.

1. Drain the filling solution from the electrode.

2. Rinse the filling chamber with DI water and drain it.

3. Refill the chamber with DI water.

4. Prepare a 50 percent (w/v) NaOH solution by slowly adding 30 g of NaOH to 30 mL of DI water.

5. Gently stir the solution with up to three electrodes to dissolve the NaOH. The solution will be very hot and may boil and splatter; caution must be used.

6. Stir the solution an additional 2 minutes with the electrodes.

7. Rinse the electrodes in DI water.

8. Rinse the electrodes in pH 7.00 buffer for 2 minutes.

9. Drain the DI water from the filling chambers.

10. Refill each electrode with 3 M KCL, agitate the electrodes, and drain the chambers.

11. Refill the chambers once more with 3 M KCL and spin each electrode from the leader to remove air bubbles.

5.4 Preparing Soil Suspensions

1. Weigh 20.0 ± 1 gram of air-dry mineral soil into a beaker and add 20.0 mL of DI water; for organic soils, use 5.0 g of soil and 25.0 mL of DI water. Log exact sample weight [4-B] and volume [4-C].

2. Allow soil to absorb the liquid without stirring.

3. Stir the mixture for 10 seconds and allow mixture to sit for 15 minutes.

4. Repeat Step 3 three times.

5. Repeat steps 1 through 4, replacing the DI water with 0.01 M $CaCl_2$. Log sample weight [5-B] and volume of 0.01 M $CaCl_2$ [5-C].

5.4.1 pH Measurements

1. After the final stirring, allow the suspension to settle for at least 1 minute. Place the pH electrode in the supernatant of the soil suspension.

   For mineral soils, the reference junction should be below the solution surface and above the soil-solution interface.

   Some organic soils swell upon wetting, so there is no free water available. As long as the reference junction is below the surface of the organic material, an acceptable, repeatable reading generally is attained. When the reading is stable, record pH to the nearest 0.01 pH unit.

2. Report the pH of the soil:DI water suspension, [4-D] and the soil:0.01 M $CaCl_2$ suspension [5-D], for each sample.

3. After measurements are completed, store the electrode in 0.1 M KCl storage solution. Do not let the sensing element and reference junction dry out. The level of the storage solution should be one inch below the filling solution level to prevent influx of the storage solution. Check periodically that the electrode reservoir is full of filling solution.

5.5 Calculations

No calculations are required to obtain pH values. Replicate measurements of the same sample or of duplicate samples should not be averaged.

6.0 QUALITY CONTROLS

Log all quality control (QC) data on appropriate form (Attachments 10.3 and 10.4).

Reagent Blanks–Analyze one blank of each suspension solution. The blank used for each pH method is the reagent used: DI water or 0.01 M $CaCl_2$. The measured pH of each blank should fall between 4.5 and 7.5 pH units.

Replicates–One sample from each batch of ten samples should be analyzed in triplicate for pH in each of the DI water and 0.01 M $CaCl_2$ solutions. The standard deviation for each set of three replicates should be 0.10 pH units or less. If fewer than ten samples are run in one day, then one sample processed that day should be analyzed in triplicate.

Quality Control Check Sample (QCCS)–A pH 7.00 standard from a different preparation source or lot number than that used for the calibration is used as the QCCS. Analyze a QCCS before beginning analysis of routine samples, at specified intervals thereafter, e.g., after every ten samples and after completion of routine sample analysis for the day. Measured values of each QCCS should be 7.00 ± 0.05. If the QCCS does not meet this criterion, recalibrate the electrode and repeat the QCCS measurement using a fresh QCCS. If acceptable results still cannot be obtained: check electrode for clean reference junction, check wiring straps into meter, check for static electricity, and check to see if enough filling solution is contained within the electrode. If a problem still persists, replace electrode or meter, or both.

QC Audit Sample (QCAS)–The QCAS (provided by the quality assurance [QA] manager) should fall within the ± 0.05 unit accuracy window.

7.0 ENVIRONMENT, SAFETY AND HEALTH

Wear protective clothing (laboratory coat and gloves) and safety glasses when preparing reagents, especially when concentrated acids and bases are used. The use of concentrated acids should be restricted to a fume hood. Always add acid to water.

8.0 RECORDS

The following documents will be maintained as Quality Records.

- Field logbook
- "Parameter - pH_H_2O, pH in water" form
- "Parameter - pH_01M, pH in 0.01M Calcium Chloride" form
- "Parameter - pH_H_2O, pH in water" QC form
- "Parameter - pH_01M, pH in 0.01M Calcium Chloride" QC form

9.0 REFERENCES

American Society for Testing and Materials. 1984. *Annual Book of ASTM Standards Vol. 11.01, Standard Specification for Reagent Water,* D-1193-77 (reapproved 1983). ASTM, Philadelphia, Pennsylvania.

Orion Research Incorporated. 1983. *Institutional Manual - Model 611 pH/millivolt meter.* Orion, Cambridge Massachusetts.

U.S. Department of Agriculture/Soil Conservation Service. 1984. *Soil Survey Laboratory Methods and Procedures for Collecting Soil Samples.* Soil Survey Investigations Report No. 1, U.S. Department of Agriculture, U.S. Government Printing Office, Washington, D.C.

10.0 ATTACHMENTS

Attachment 10.1 "Parameter - pH_H2O, pH in water" form

Attachment 10.2 "Parameter - pH_01M, pH in 0.01M Calcium Chloride" form

Attachment 10.3 "Parameter - pH_H2O, pH in water" QC form

Attachment 10.4 "Parameter - pH_01M, pH in 0.01M Calcium Chloride" QC form

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Parameter - PH _ 01M
pH in 0.01M Calcium Chloride

Batch # _____
Lab Code _____

Submission # _____
Run # _____
Re-Analysis _____
Operator Initials _____

Date Analysis Started ___/___/___
Date Analysis Completed ___/___/___
Date Form Completed ___/___/___
Lab Manager Initials ____

A Samp. ●	B Sample Weight (g) XX.X	C Solution Volume (mL) XX.X	D Result (pH) X.XX
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			
15			
16			
17			
18			
19			
20			
RBLK1	XXXXXXXXXXXXXXXXXXXX	XXXXXXXXXXXXXXXXXXXX	

AWC

Parameter - PH _ H_2O
pH in Water

Batch # _____
Lab Code _____

Submission # _____
Run # _____
Re-Analysis _____
Operator Initials _____

Date Analysis Started ___/___/___
Date Analysis Completed ___/___/___
Date Form Completed ___/___/___
Lab Manager Initials ____

REPLICATES				
A Samp. # Used XX	B Sample Wt. (g) XX.X	C Solution Vol. (mL) XX.X	D Result (pH) X.XX	E SD XXX.XX

QCCS	(pH) X.XX
True	
High	
Low	
QCCS 1	
QCCS 2	
QCCS 3	
QCCS 4	
QCCS 5	
QCCS 6	
QCCS 7	
QCCS 8	

AWC

Parameter - PH _ .01M
pH in 0.01M Calcium Chloride

Batch # _____
Lab Code _____

Submission # _____
Run # _____
Re-Analysis _____
Operator Initials _____

Date Analysis Started ___/___/___
Date Analysis Completed ___/___/___
Date Form Completed ___/___/___
Lab Manager Initials ____

REPLICATES				
A Samp. # Used XX	B Sample Wt. (g) XX.X	C Solution Vol. (mL) XX.X	D Result (pH) X.XX	E SD XXX.XX

QCCS	(pH) X.XX
True	
High	
Low	
QCCS 1	
QCCS 2	
QCCS 3	
QCCS 4	
QCCS 5	
QCCS 6	
QCCS 7	
QCCS 8	

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ANALYTICAL PROCEDURES
AIR - DRY MOISTURE DETERMINATION

Approvals:

Terry Wenstrand	5/2/91	Gregory Fadie	5/2/91
Project Manager	Date	Radiation Safety Officer	Date
Gregory Fadie	5/2/91	Gregory Fadie	5/2/91
QA Manager	Date	E,S&H Coordinator	Date

1.0 PURPOSE AND SCOPE

The purpose of this analytical procedure (AP) is to instruct laboratory personnel in the methods used for determining the moisture content of soil samples.

2.0 APPLICABILITY

This AP applies to laboratory personnel involved in soil analysis activities in which the moisture content of soil samples must be determined.

Air-dry moisture determination is accomplished at the analytical laboratory. The procedure for assessing air-dry moisture is not to be performed, however, until the soil is believed to be air dry (see section 5.4). Initially, the process is used to ensure that each sample is at an acceptable moisture level for further processing, after which, the air-dry moisture is determined on all samples to convert all results to an oven-dry basis, and if specified in a procedure, to calculate the weight of sample equivalent to a given weight of oven-dry soil (Brady, 1974).

A subsample of the air-dried bulk soil sample is weighed, oven-dried for approximately 24 hours, and reweighed. The initial and final weights are used to calculate a percent weight loss.

3.0 DEFINITIONS

4.0 RESPONSIBILITIES

The laboratory supervisor is responsible for assuring that all personnel assigned a task related to this AP are familiar with this AP, that they have been trained in the AP, and that they have a controlled copy of this AP.

All laboratory personnel are responsible for following this AP.

5.0 PROCEDURE

Interferences

Sample weights can be affected by salts, oils, and moisture present on skin. Losses of fine silt and clay, caused by excessive movement of the samples, will cause erroneously calculated moisture values.

CAUTION: If a sample is suspected to contain radionuclides, workers must wear impermeable rubber gloves when handling the sample.

5.1 Sample Collection, Preservation, and Storage and Aliquot Preparation

A subsample is taken from the bulk soil sample after it is believed to be fully air-dried. Analysis takes place immediately thereafter; no preservation or storage is required.

5.2 Equipment and Supplies

5.2.1 Apparatus

1. Balance, capable of weighing to 0.01 g.
2. Drying or Convection oven.
3. Desiccator oven.
4. Forceps or impermeable rubber gloves.
5. Thermometer, 0°C to 200°C range.
6. Weighing containers capable of withstanding intermittent heating to 110°C and cooling to room temperature.
7. Balance calibration weights, 3-5 weights covering expected range.

5.2.2 Consumable Materials

1. Aluminum weighing dishes, pre-numbered.

2. Aluminum weighing pans, disposable.

3. Gloves, powdered.

4. Air-dry moisture raw data forms (Attachment 10.2) and air-dry moisture log book.

5. Form 1, air-dry moisture percent, and form QC-1 (Attachment 10.3 and 10.4, respectively).

5.3 Calibration and Standardization

The standardization of the balance is described in AP 1, General Laboratory Procedures. The thermometers should be checked monthly against an NBS traceable standard thermometer to ensure that they are measuring temperature accurately. The oven should be monitored to ensure that temperature fluctuation does not exceed ± 10° C.

5.4 Sample Drying

1. Label a bulk sample processing raw data form (see Attachment 10.1) for each sample to be air dried.

2. Place two fresh sheets of kraft paper, approximately 1m x 1m in area, on the mesh partition of the drying table. With gloved hands, slowly spread the sample on top of the sheets of paper, taking care not to lose any soil off the paper or contaminate any adjacent samples.

   CAUTION: Any sample suspected to contain radionuclides must be processed in an approved, ventilated area such as a fume hood.

   NOTE: In some cases, samples may be received from the field in more than one sample container (bag). Combine the contents of all containers comprising a single sample.

3. Desegregate any large aggregates that impede the spreading of the sample over the entire area of the paper. Place an additional sheet of kraft paper loosely over the sample.

4. Identify the sample by attaching the sample's processing raw data form to a hook attached to the drying table or other mechanism. The original canvas and plastic sampling bags should be kept on the floor beneath the sample as a second check on sample identity.

   NOTE: Immediately after a sample is spread to dry, field-moist pH is determined (see AP 4).

5. Daily, stir the soil sample to facilitate drying.

6. For the first few days that a wet sample is spread, the bottom sheet of paper may need to be changed daily in order to alleviate excessive moisture accumulation. Any observation of fungal or algal growth should be noted on the bulk sample processing raw data form.

NOTE: Soils high in clay may harden nearly irreversibly if allowed to dry without a preliminary disaggregation of medium and coarse pedons. An effort should be made during the air-drying procedure to desegregate these pedons by physical manipulation with gloved hands while still somewhat moist or friable, before reaching an air-dry state.

7. Allow the sample to air dry until it is believed to be at or below the specified moisture content. This process generally takes about two days, although drying time may vary from one day to a week or more.

8. Subsample an aliquot for the air-dry moisture determination.

5.5 Laboratory

NOTE: Data for this procedure are recorded on the air-dry moisture raw data form (see Attachment 10.2). Once a soil sample is determined to be air dry, the information is also entered on the bulk sample processing raw data form (Attachment 10.1).

1. Thoroughly mix the air-dry sample. Transfer a subsample of approximately 15 g into a pre-numbered aluminum weighing dish of known weight. Enter the dish number on the air-dry moisture raw data form under "TIN_NO." Carefully handle the weighing dish by its edges or with forceps. If the sample is suspected to contain radionuclides wear impermeable rubber gloves when handling the dish directly.

NOTE: Because the aluminum weighing dishes or equivalent are manufactured to be a nearly constant weight, the average weight of ten labeled aluminum weighing dishes may be used.

2. Weigh the dish plus sample to the nearest 0.01 g and record this initial weight under "INIT_WT." Place the dish in a convection oven which has equilibrated at 105° C for mineral samples or 60° C for organic samples. Allow the sample to oven dry overnight at this temperature.

3. The following morning, remove the sample from the oven and allow to cool for 30 minutes in desiccator. Weigh the dish plus sample to the nearest 0.01 g and record this oven dry weight under "OD_WT."

NOTE: Data for this procedure are recorded on form 1 oven dried moisture percent (Attachment 10.3). Figures within the square brackets, [ ], represent the form number and column in which the data are recorded.

4. Remove weighing pan from oven; allow soil to cool in a desiccator. Weigh each sample to ± 0.01 g and record oven-dry weight [1-C].

5.6 Calculations

The following calculations may be performed by computer. It is advantageous to manually check a few samples using these calculations to understand the procedure and to test the accuracy of the computer program.

$$\textit{PERCENT AIR-DRY MOISTURE (MOIST P)} = \frac{\textit{INIT WT - OD WT}}{\textit{OD WT - TIN WT}} \times 100$$

If the calculated moisture content is above 2.5 percent for mineral soils or above 6.0 percent for organic soils, allow the bulk sample to continue air-drying and repeat the procedure at a later time. However, if the sample is below the cutoff percent moisture content for the soil type, then the air-dry sample may be rebagged and placed in cold storage or it may immediately undergo the next stage of processing.

NOTE: Designations within the square brackets, [], represent the form number and column in which the data are recorded on Form 1 (Attachment 10.3).

Percent moisture = ([Air-dry wt. - Oven-dry wt.] ÷ Oven-dry wt.) x 100

OR

MOIST [1-D] = ([1-B] - [1-C]) ÷ [1-C] x 100

6.0 QUALITY CONTROLS

NOTE: Analytical laboratory quality control (QC) results are recorded on Form QC-1 (Attachment 10.4).

Replicates--One sample from each batch of ten samples or less per day should be analyzed in duplicate. The percent relative percent difference (%RPD) should be 15 percent or less.

QC Audit Sample (QCAS)--The QCAS should fall within the accuracy window provided by the quality assurance (QA) manager.

7.0 ENVIRONMENT, HEALTH AND SAFETY

Forceps or heat-resistant gloves should be used to handle weighing dishes after removal from the oven. If samples are suspected to contain radionuclides impermeable rubber gloves must be worn when directly handling the weighing dishes.

8.0 RECORDS

The following documents will be maintained as Quality Assurance records.

- Bulk sample raw data
- Soil sample air dry determination
- Parameter moist air dry moisture percent
- QC Form parameter moist air dry moisture percent

- Field logbook
- "Bulk Sample Raw Data" form
- "Soil Sample Air Dry Determination" form
- "Parameter-Moist, Air Dry Moisture Percent" form
- "Parameter-Moist, Air Dry Moisture Percent" form

9.0 REFERENCES

Brady, N.C. (ed.). 1974. The Nature and Property of Soils. Eighth Edition. MacMillan Publishing Co., Inc., New York, New York.

10.0 ATTACHMENTS

Attachment 10.1 "Bulk Sample Raw Data" form

Attachment 10.2 "Soil Sample Air Dry Determination" form

Attachment 10.3 "Parameter-Moist, Air Dry Moisture Percent" form

Attachment 10.4 "Parameter-Moist, Air Dry Moisture Percent" QC form

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BULK SAMPLE RAW DATA

SAMPLE ID:_____ DATE SAMPLED: ___/___/___

SITE ID:_____ DATE RECEIVED: ___/___/___

SET ID:_____ PROCESS START: ___/___/___

BATCH ID:_____ PROCESS COMPLETE:___/___/___

SOIL TYPE: M / O FIELD pH: _____

Initials: _____ Initials: _____

AIR SAMPLE DRYING:

Date	% Moisture	Initials
___/___	_____	_____
___/___	_____	_____
___/___	_____	_____

TOTAL BULK WT:

Date: ___/___/___ _____.____ g Initials:_____

ROCK FRAGMENT WT:

2 TO 4.75 mm: _____.____ g 4.75 to 20 mm: _____.____ g

Date: ___/___/___ Initials: _____

ENTERED IN COMPUTER: Date: ___/___/___ Initials: _____

COMMENTS:

_____ _____

Supervisor Date

SOIL SAMPLE AIR DRY DETERMINATION

ALL WEIGHTS REPORTED TO 0.01 GRAMS

SAMPLE CODE	TIN NO.	TIN WT.	AIR DRY (TIN+SOIL)	OVEN DRY (TIN+SOIL)	DATE	ID	COMMENTS	OPERATOR INITIALS	DATE

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Attachment 10.3
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ANALYTICAL PROCEDURES
AIR - DRY MOISTURE DETERMINATION

Parameter - Moist
Air Dry Moisture Percent

Batch # _____
Lab Code _____

Batch # _____
Submission # _____
Run # _____
Re-Analysis _____
Operator Initials _____

Lab Code _____
Date Analysis Started ___/___/___
Date Analysis Completed ___/___/___
Date Form Completed ___/___/___
Lab Supervisor Initials ____

A Samp. #	B Air Dried Sample Wt. (g) XX.XX	C Oven Dried Sample Wt. (g) XX.XX	D Calculated Result (%) XX.XX
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			
15			
16			
17			
18			
19			
20			

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Parameter - Moist
Air Dry Moisture Percent

Batch # _____
Lab Code _____

Batch # _____
Submission # _____
Run # _____
Re-Analysis _____
Operator Initials _____

Lab Code _____
Date Analysis Started ___/___/___
Date Analysis Completed ___/___/___
Date Form Completed ___/___/___
Lab Supervisor Initials ____

REPLICATES				
A Samp. #	B Air Dried Sample Wt. (g) XX.XX	C Oven Dried Sample Wt. (g) XX.XX	D Calculated Result (%) XX.XX	E SD or %RSD XXX.XX