

The Arizona Border Study

*An Extension of the
Arizona National Human Exposure Assessment Survey (NHEXAS) Study
Sponsored by the Environmental Health Workgroup of the Border XXI Program*

Quality Systems and Implementation Plan for Human Exposure Assessment

The University of Arizona
Tucson, Arizona 85721

Cooperative Agreement CR 824719

Standard Operating Procedure

SOP-UA-L-11.1

Title: Soil Characterization

Source: The University of Arizona

U.S. Environmental Protection Agency
Office of Research and Development
Human Exposure & Atmospheric Sciences Division
Exposure & Dose Research Branch

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Title: SOIL CHARACTERIZATION

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APPROVALS

☒ Full SOP ☐ Working SOP #pages 5

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Soil Characterization

1.0 Purpose and Applicability

The purpose of this SOP is to describe the procedures to be followed in splitting, determining the grain size characteristics, electrical conductivity (EC), and pH of the "Composite Soil" and "Foundation Soil" samples. This procedure applies to the general characterization of sediment of the soil samples for the EPA NHEXAS project of the University of Arizona/Battelle/Illinois Institute of Technology consortia, as well as future "Health in the Environment" investigations.

2.0 Definitions

- 2.1 DDW = Deionized Distilled Water
- 2.2 Soil = Sediment collected during the project from the yard and along the foundation of participants houses.

3.0 References

- 3.1 Folk, Robert L. 1980. *Petrology of Sedimentary Rocks*, Hemphill Publishing, Austin Texas. pp. 184.
- 3.2 Soil Survey Staff. 1951. *Soil Survey Manual*, United States Department of Agriculture, Washington D.C. pp. 503.

4.0 Discussion

During the weathering of sediment differential loss of mineral components occur as grain size is reduced. Soil samples must be split to facilitate analysis in separate laboratories. In splitting the samples each portion must be an accurate representation, in its distribution of different particle sizes, of the whole. Further characterization of the soil samples will allow analysis of the soil grain size with other more intensive soil analyses. The remaining samples are split into defined size categories using soil sieves. Weight determination on each sample will be used to determine the weight percent of each size class. A reading of the pH and conductivity (EC) will be taken as a measure of the weathering environment and free ions.

5.0 Responsibilities

- 5.1 The Project Director will be responsible for;
 - 5.1.1 final review and approval of this procedure.
- 5.2 The Project Lab Supervisor will be responsible for;
 - 5.2.1 insuring SOP procedures are followed by the Project Lab Staff,
 - 5.2.2 notifying the appropriate technicians with needed repairs. In cases when the ite can not be fixed in-house, Project Field Coordinator will generate the appropriate paperwork, notify the appropriate vendor or company, and ship the dysfunctional

item.

- 5.3 The Project Lab Staff will be responsible for;
- 5.3.1 knowing and following the procedures described in this SOP,
 - 5.3.2 recording the information as directed in this SOP,
 - 5.3.3 notifying the Project Lab Supervisor with down equipment and repair supplies needed (where applicable),
 - 5.3.4 providing the Project Lab Supervisor with down equipment label and isolating the down equipment into the down equipment area,
 - 5.3.5 insuring proper labeling techniques of down equipment,
 - 5.3.6 repairing the item (where applicable) in a timely manner.

6.0 Equipment and Materials

6.1 Equipment

- 6.1.1 Acculab #V1200 balance
- 6.1.2 Beaker, 100 ml
- 6.1.3 "Blue Ice" ice packs
- 6.1.4 Brass 1000 g calibration weight (#9800009)
- 6.1.5 Brass 100 g calibration weight (#9800010)
- 6.1.6 Brass 10 g calibration weight (#9800011)
- 6.1.7 Brass 5 g calibration weight (#9800017)
- 6.1.8 Brass 1 g calibration weight (#9800018)
- 6.1.9 Conductivity Meter
- 6.1.10 Conductivity Calibration Standard
- 6.1.11 Kim Wipes
- 6.1.12 Metal tray
- 6.1.13 Mettler Balance
- 6.1.14 pH paper with a range from 1 to 11 at 1.0 increments
- 6.1.15 pH paper with a range from 1 to 11 at .5 increments
- 6.1.16 Ruler
- 6.1.17 Scissors
- 6.1.18 Teflon coated tweezers
- 6.1.19 Tylor Stainless Steel Sieve, #10
- 6.1.20 Tylor Stainless Steel Sieve, #230
- 6.1.21 Weighing pans
- 6.1.22 Ziploc plastic bags

6.2 Materials

- 6.2.1 DDW (deionized distilled water)

7.0 Procedure

7.1 Preparation of work area for splitting operation

- 7.1.1 Clean the work area with DDW and paper towels.
- 7.1.2 For each sample to be processed fill out a "Soil Characterization Form" (figure 1). Prepare new plastic bags for each split by placing an "aliquot sample ID" label on the bag for the pesticide split and one of the original sample ID labels on the pH/EC split bag. Fill out a new "Chain of Custody" form (figure 2) for the pesticide split.
- 7.1.3 Set up a cold surface to work on by placing "blue ice" bags between two inverted metal trays. The tray surface provides a cold surface to reduce the amount of heating that each sample experiences during aliquoting.

7.2 Preparation of work area for sieving

- 7.2.1 Clean the work area with DDW and paper towels.
- 7.2.2 Locate the "Soil Characterization Form" (figure 1) in the "Uncompleted Sample Form" notebook for each of the samples to be processed.
- 7.2.3 Locate stacked soil sieve sets consisting of a solid pan, sieve #230, sieve #10, and a top.
- 7.2.4 Prepare the equipment needed to clean the sieves after each use (UA-L-5.1).
- 7.2.5 Turn on and calibrate the Acculab V-1200 balance (UA-L-1.1).

7.3 Preparation of work area for pH/EC readings

- 7.3.1 Clean the work area with DDW and paper towels.
- 7.3.2 Locate the 100 ml beakers, cleaned as per SOP UA-L-5.1, for the pH and EC measurements.
- 7.3.3 Locate a 100 ml graduated cylinder cleaned to specifications (UA-L-5.1).
- 7.3.4 Locate the electrical conductivity (EC) probe. It consists of a meter attached to wand that contains contact points. Once immersed into a solution the meter records the electrical conductivity in μmhos .
- 7.3.5 Set out two 100 ml beakers, cleaned as per SOP UA-L-5.1, one to hold electrical conductivity meter calibration solution and the other DDW.
- 7.3.6 To calibrate the "Electrical Conductivity Meter" insert the calibration plug, a gray plug without a cord, into the electronic outlet for the wand on top of the meter. Slide the switch on the side of the unit to "microMho". Set the reading to 1.0 by turning the calibration set screw on the top of the unit.
- 7.3.7 Shake the conductivity meter calibration solution vigorously to ensure mixing. Place 10 - 20 ml into a clean beaker. Calibration solution is very sensitive to contamination from many sources (e.g. the air, glassware surfaces, utensils, etc.). Place the probe into the solution and record the value in the "EC Calibration Log Book". After analysis of the samples the procedure is repeated. The second

reading must be within 5% of the initial for the samples to be valid. If the difference is greater than 5% the samples are considered to be not valid.

7.4 Sample Splitting

- 7.4.1 Prepare the area according to 7.1
- 7.4.2 The samples are received in a plastic Ziploc sample bag inside of a brown paper bag. The paper bag has the chain of custody record stamped onto the outside (UA-F-5.1, UA-F-6.1).
- 7.4.3 Three splits are needed from each sample (figure 3). The "Pesticide Split" needs at least 2 grams of the finest fraction for processing at other laboratories, therefore from 1/3 to 1/4 of the total is split from the original sample. In addition, approximately 10 g are separated for the "pH and Electrical Conductivity" split. The remainder is retained for soil characterization and metals testing.
- 7.4.4 Mix and homogenize the soil sample within its original plastic sample bag. Place the sample bag on to the cold work surface, on top of a large kim wipe.
- 7.4.5 Slip a ruler underneath the sample bag. Lift the ruler and turn on edge causing the sediment to fall to each side, separating 1/3 to 1/4 of the total from the original sample. Cut along the ruler creating the pesticide split. Using the cut bag as a scoop place the material into the pre-labeled "pesticide" sample bag. Return the pesticide split to the freezer.
- 7.4.6 Repeat procedure 7.4.5 on the opposite corner of the original sample bag to separate approximately 10 grams for the pH/EC split. Cut along the ruler creating the split. Using the bag as a scoop place the material into the prelabeled "pH/EC" sample bag. Place the pH/EC split sample back into the original paper bag to await analysis.
- 7.4.7 Analysis techniques do not require the pH/EC split to be dry, however soil characterization is hampered if the sediment is damp. Open the "Soil Characterization split" bag by rolling over the top, then return it to the original paper bag. The top of the paper bag is folded over and secured. The "Soil Characterization Split" is stored at room temperature until dry (figure 4). Record the date and time that drying starts under 2 "Drying Time" (figure 1).
- 7.4.8 Repeat steps 7.4.4 through 7.4.9 for each sample..

7.5 Pesticide Split

- 7.5.1 Pesticide splits are placed into the freezer in the laboratory (room 130A) once they are separated from the original samples.
- 7.5.2 No further processing occurs at the U of A lab.
- 7.5.3 Pesticide splits are transferred to the Materials Tech and maintained at freezer temperatures until shipment to other laboratories to detail the pesticide content.

7.6 pH/EC measurement and color determination of the "pH and Electrical Conductivity aliquot".

- 7.6.1 Remove the "pH/EC split" sample from the original sample paper bag.
- 7.6.2 Use the Munsell Soil Color Chart to identify the color of the pH sample, and record on the "Soil Characterization form" under #3 "pH and Conductivity" subsection F "color".
- 7.6.3 Take approximately 10 grams from the pH/EC split and place in a pre-weighed and tared plastic weighing boat. Weigh the sample on the Acculab #V-1200 and enter the weight on the "Soil Characterization form" (figure 1) under #3 "pH and Conductivity", subsection B "weight".
- 7.6.4 Chose a 100 ml beaker and mark the Sample ID on the side with a grease pencil.
- 7.6.5 Measure into a graduated cylinder, four times the pH/EC sample aliquot weight in ml of DDW and place in the beaker. Record the amount of water on the "Soil Characterization form" (figure 1) under #3 "pH and Conductivity", subsection C, "H₂O Added".
- 7.6.6 Place the soil pH/EC sample in the beaker.
- 7.6.7 Swirl the mixture for approximately 30 seconds.
- 7.6.8 Using a pair of tweezers, dip a piece of pH paper with the broadest range into the solution. Determine the pH by reading the color scale on the side of the pH paper container. Repeat the procedure using a narrower range pH paper which will allow readings to 0.5 of a number.
- 7.6.9 Record the readings from the pH paper on the "Soil Characterization" form (figure 1) under #3 "pH and Conductivity", subsection D, "pH".
- 7.6.10 Place the EC conductivity wand into the sample beaker. Allow to stabilize and record on the "Soil Characterization form" (figure 1) under #3 "pH and Conductivity", subsection E, "Conductivity". If the measurements fall outside of the range in μ mhos, place the switch at the side of the meter to megohms and re-measure. Microhms are calculated by taking the inverse of the mesured megohms. Record the μ mho calculation on the "Soil Characterization Form".
- 7.6.11 Rinse the wand with distilled water until a reading equivalent to sterile DDW is achieved.
- 7.6.12 Discard the pH/EC sample mixture.
- 7.6.13 Repeat steps 7.6.1. to 7.6.12. for each sample.

7.7 "Soil Characterization Sample" Sieving

- 7.7.1 Moisture in a sample can hamper efficient sieving, thus a period of air drying is necessary. The plastic bag containing the sample is placed in the outer paper sample bag after the splitting process (7.4.9). Dry at room temperature until dry, or for at least 72 hours (figure 4).
- 7.7.2 Remove the sample from the paper bag. If the sample appears moist return it to its

paper bag for further drying. Desegregate the sample using manual manipulation through the plastic bag. Enter the date and time on the "Soil Characterization" form (figure 1) under 2 "Drying Time", finish.

- 7.7.3 Mark the size fraction and sample number on weighing pans for each of the fractions. Weigh the weighing pans and enter the weight into 5 "Particle Size" area on the "Soil Characterization" form (figure 1) under #2, #5 and #8. Record the scale to be used to weigh the two large fractions and the fine fraction (figure 1).
- 7.7.4 Pour the desegregated sample onto the top of a stack of soil sieves, which decrease in screen size (#10, #230, and solid pan) from top to bottom.
- 7.7.5 Agitate the sieves, inside the fume hood, for 1-2 minutes.
- 7.7.6 Carefully concentrate the particles along one edge by tipping the sieves during the last few agitations. End the sieving with a couple of firm strikes to the side of the sieves to loosen any residual particles from the screen surfaces.
- 7.7.7 Gently tap each particle size fraction into pre-weighed (7.7.3), pre-labeled weighing pans and weigh as outlined below in 7.8.
- 7.7.8 Place the sieves in the sink for cleaning (UA-L-5.1). Sieves are cleaned between each sample.
- 7.7.9 Repeat steps 7.7.1 to 7.7.8 for each sample.

7.8 "Soil Characterization" Sample Weighing of Sieved Fractions

- 7.8.1 Weigh and record each fraction weight on the "Soil Characterization" form (figure 1) under 4 "Particle Size" as weight #1, #4, and #7. In each case the total wt. (#1, #4, #7) minus the weighing pan (#2, #5, and #8) is the sample wt. (#3, #6, and #9). Weigh the standard weight (#9800010, #9800011, #9800017, #9800018) closest to the weight of the material weighed on each of the scales. Record the ID# and weight under "4 Standard Weights" on the "Soil Characterization" form.
- 7.8.2 Transfer the two largest particle subsamples (>#10 and #10-230) to small plastic bags. Label with the sample ID# and place into the paper sample bag for archival.
- 7.8.3 The fine fraction is analyzed using the XRF (UA-L-10.1) then archived or transferred to other laboratories for further analysis. From 1 to 4 grams of material is needed and can easily fit into a plastic XRF cup. Once the fine fraction is weighed in the weighing pan, pour all but approximately 1 to 4 grams into a ziplock bag, label with the sample ID# and place into the paper sample bag. The remaining material is placed back on the scale and the weight recorded under #9 as the XRF Sample weight. The weighing pan weight is the same as stated in 7.8.1. The "XRF sample total weight (#9)" minus the "weighing paper wt. (#11)" is the "XRF sample weight (#12)". Transfer the material into an XRF cup and record the number onto the "Soil Characterization Form" (figure #1) and the "XRF Analysis Form" (figure #5). A Chain of Custody form is filled out and the appropriate label

assigned for the split.

- 7.8.4 Place X-ray mylar film over the top of the XRF cup and secure with a plastic side ring. Fit the top ring onto the cup ensuring that the mylar film is stretched tight and flat. Record the cup number on the "Soil Characterization" form (figure 1) under "#5 Fine Fraction Split" and fill out the header information on an "XRF Analysis" form (figure #5).
- 7.8.5 Place the XRF cup into a sample tray and place onto one of the stacked shelves next to the XRF (see SOP UA-L-10.1). Place the "XRF Analysis" form (figure #5) onto the top of the sample tray.
- 7.8.6 After XRF analysis, pour the sample into a small plastic bag. Label with the sample ID#. Archive (room 130B) with the other size fractions or ship to another laboratory for further analysis.
- 7.8.7 Repeat procedures 7.8.1 through 7.8.6 for each sample.

7.9 Calculations

$\Sigma(\text{Particle Sample Weights \#3, \#6, and \#9}) = \text{Total Sieved Weight (\#10)}$

$<230 \text{ Sample Wt. (\#9)} - \text{Remaining Wt. (\#11)} = \text{XRF Sample Wt. (\#12)}$

$1 / \text{megohm} = \text{micromho} \qquad 1 / \text{micromho} = \text{megohm}$

7.10 Quality Control

7.10.1 Tolerance Limits

A. Weighing

1. Weighing will occur on the balance in the Laboratory (Room 130A) and will follow the standard operating protocol for the "Calibration and Operation of NHEXAS Balances" (UA-L-1.1).

B. pH/EC readings

1. The pH paper is read to the nearest 1 pH unit to choose the finer scaled pH paper to use. The finer scaled pH paper is read to the nearest 0.5.
2. The electrical conductivity probe is read to the nearest tenth. After each reading the probe is rinsed with DDW until it reflects the EC of the DDW. The wand is placed in a calibration solution with a known conductivity prior to analysis and after the measurements are taken. The reading must be within 5% of each other for the measurements to be valid. Failure to meet the calibration criteria after two attempted readings results in the loss of all data.

7.10.2 Detection Limits

A. pH Paper

1. The pH paper has a range of 1 to 11 at the coarse grade. Once the range is determined pH paper with a finer scale, 0.5, is used to determine the pH level.

B. Electrical conductivity Meter

1. The electrical conductivity meter reads values from 0.1 to 199.9 μmhos . The meter automatically compensates for temperatures to the international standard of 25 °C. The temperature compensation range is from 0.0 to 50.0 °C (32.0 to 122.0 °F).

C. Sieve Screens

1. Each of the sieve screens is certified to be accurate to ASTM E11, ANSI, and ISO specifications (see figure #6 for an example of the Certificate of Authentication kept on file for each soil sieve) .

7.9.3 Corrective Actions

A. Electrical Conductivity Meter

1. If the conductivity meter fails calibration and/or to meet quality control criteria twice the unit is tagged and returned to the manufacturer for service.

8.0 Records

8.1 Data Collected by this Procedure

- 8.1.1 Each of the measurements will be recorded onto the "Soil Characterization Form" (L-11.0-1.0; figure #1) as they are made.

8.2 Location/Placement of Forms

- 8.2.1 The "Soil Characterization" form records a number of activities occurring over some period of time. Until all activities performed on the sample are completed, the "Soil Characterization" form (L-11.0-1.0; figure #1) will be kept in the Laboratory (Room 130A) in the "Uncompleted Sample Form" notebook. Once completed the forms are transferred to the Data Coordinator.
- 8.2.2 XRF Analysis forms (L-10.0-1.0) corresponding to loaded sample cups will be kept on top of the sample tray containing the samples. The sample trays are placed on one of the stacked shelves to await XRF analysis. The XRF is placed in the laboratory (room 130A). Once completed the XRF forms are filed in the laboratory supervisors office as a back-up to electronically generated files of the data.
- 8.2.3 A sign-out sheet is kept for notebooks and files. Any individual removing a notebook or file records their name and date of removal on the sign-out sheet, located in the Lab Supervisor's office.

Figure 1. Soil Characterization Form

SOIL CHARACTERIZATION FORM

Technician: _____ [][] Start Date ____/____/____

Init. Code

Sample ID: [][][][][][][][] HHID: [][][][][][][][] FS: [] Status: []

#1 Pesticide Split;	Aliquot ID#: [][][][][][][][]	Status Code []	0
#2 Drying Time	Start [][]:[][] Date: ____/____/____		0
	Finish [][]:[][] Date: ____/____/____		0
#3 pH and Conductivity; Tech: _____ [][]		Scale	0
A. Status ..[]	D. pH.....	0 AE163	0
B. Weight:.. _____ g	E. Conductivity _____ μ mho	0 AE166	0
C. H ₂ O:..... _____ ml	F. Color	0 V-1200	0
#4 Particle Size; Tech ID: _____ [][]			
Size	Weights:		
>10	Total Wt. _____ g - (#2) _____ g = (#3) _____ g	Scale	0
		0 AE163	0
		0 AE166	0
10-230	(#4) _____ g - (#5) _____ g = (#6) _____ g	0 V1200	0
<230	(#7) _____ g - (#8) _____ g = (#9) _____ g	Scale	0
		0 AE163	0
XRF Samp	(#9) _____ g - (#11) _____ g = (#12) _____ g	0 AE166	0
		0 V1200	0
Total Sieved Weight: (#10) _____ g			0
#5 Fine Fraction Split:	Aliquot ID#:[][][][][][][][]	Status Code []	Y N 0
	XRF Cup #:[][][]	XRF Form Header Completed [][]	0
#6 Standard Weight #:	Weight: _____ g		0
	Weight: _____ g		0

QC Initial _____ Date ____ \ ____ \ ____ \

L-11.0-1.0

[illegible]

Figure 3. Diagram detailing the splitting procedure from the original sample.

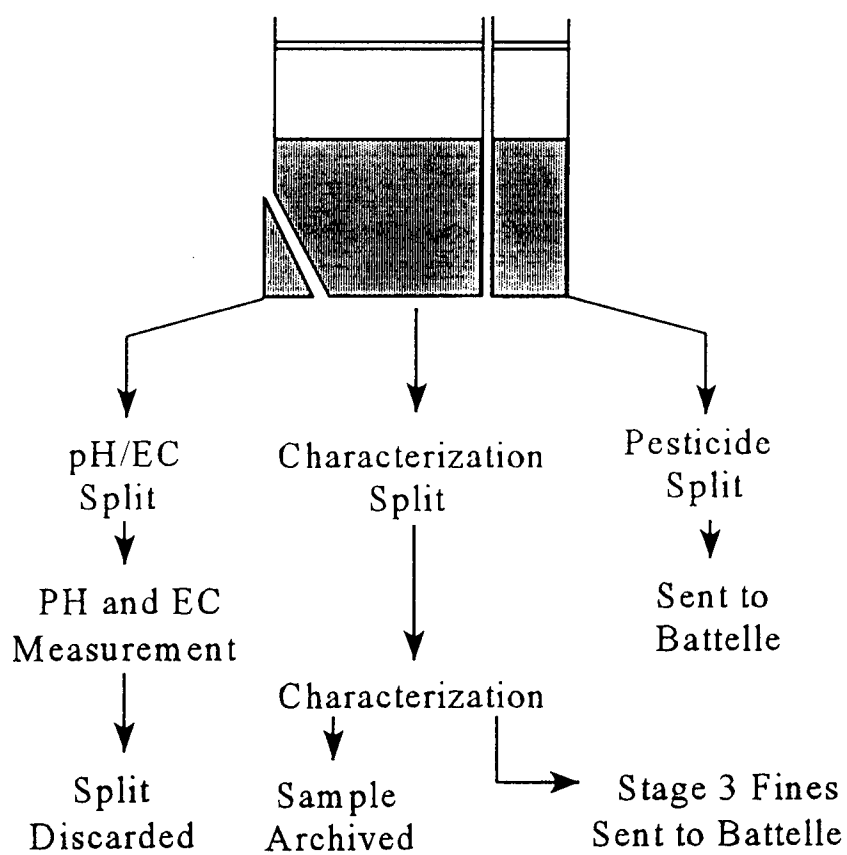


Figure 4. Graph of the % water loss per 24 hours during the drying process for 10 samples of varying weights. The time needed to ensure a dry sample that is easily sieved varies and is dependent upon a number of factors (i.e. the initial quantity of material, grain size, initial wetness, etc.)

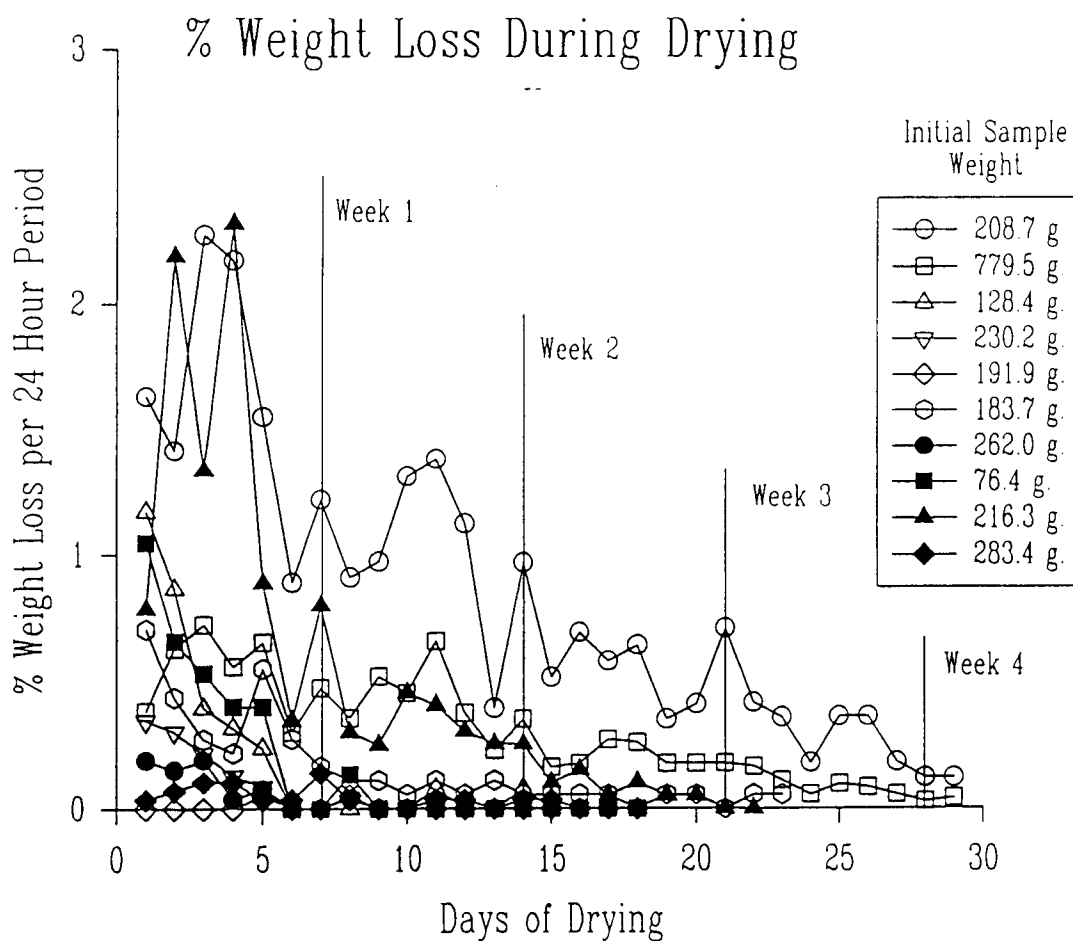


Figure 5. XRF analysis Form.

XRF ANALYSIS

Technician Init. _____ Code [][] Date ____/____/____ Time [][]:[][]

Sample ID# _____ Source Time: Fe-55 [][][]sec. Application: Thin Film []
[][][][][][][][] Cd-109 [][][]sec. Soils []
Am-241 [][][]sec.

Element	Reading ($\mu\text{g}/\text{cm}^2$; ppm)	Standard Deviation
Pb	[][][][][][][][]	[][][][][][][][]
As	[][][][][][][][]	[][][][][][][][]
Cd	[][][][][][][][]	[][][][][][][][]
Ni	[][][][][][][][]	[][][][][][][][]
Cr	[][][][][][][][]	[][][][][][][][]
Ba	[][][][][][][][]	[][][][][][][][]
Mn	[][][][][][][][]	[][][][][][][][]
Se	[][][][][][][][]	[][][][][][][][]
V	[][][][][][][][]	[][][][][][][][]
Cu	[][][][][][][][]	[][][][][][][][]
Zn	[][][][][][][][]	[][][][][][][][]
K	[][][][][][][][]	[][][][][][][][]
Ca	[][][][][][][][]	[][][][][][][][]
Co	[][][][][][][][]	[][][][][][][][]
Fe	[][][][][][][][]	[][][][][][][][]
Mo	[][][][][][][][]	[][][][][][][][]
Tl	[][][][][][][][]	[][][][][][][][]
Ag	[][][][][][][][]	[][][][][][][][]
Sr	[][][][][][][][]	[][][][][][][][]
U	[][][][][][][][]	[][][][][][][][]
Th	[][][][][][][][]	[][][][][][][][]
Sn	[][][][][][][][]	[][][][][][][][]
W	[][][][][][][][]	[][][][][][][][]
Ti	[][][][][][][][]	[][][][][][][][]
Rb	[][][][][][][][]	[][][][][][][][]
Ir	[][][][][][][][]	[][][][][][][][]
Hg	[][][][][][][][]	[][][][][][][][]

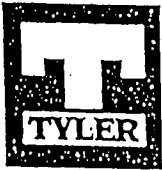
QA/QC Signature: _____ [][]

Figure 6. Authentication certificate for Tyler soil sieve # 9468400.

Certificate of Authentication

**TEST
SIEVE**

Made By

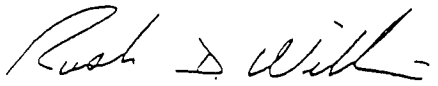


W.S. TYLER
8570 Tyler Boulevard, Mentor, Ohio 44060 USA

Specifications: ☐ **ASTM E 11** ☐ **ANSI** ☐ **ISO**

This is to certify that the test sieve
below has been submitted for analysis
in our laboratories for conformance
with the specifications set out above.

Date 10

By 
Senior Technician, W.S. Tyler

SERIAL N^o 9468400