1.0 TITLE

Saturated Paste Extraction Procedure for Soils and Soil Overburden

2.0 LOCATION

Soils Digestion and Prep Labs

3.0 SCOPE & APPLICATION

The salt concentrated in the soil solution affects plants that grow in saline soils. The principle cations present are calcium, magnesium, and sodium with small amounts of potassium. If the proportion of sodium is high, the alkali hazard is high. By making a soil-water extract and measuring the salt concentration of the extract, the salinity hazard of the soil can be determined

The saturated paste extraction is performed primarily on agronomic soils and overburden samples from coal mining operations. The moisture content of the paste is determined thus providing the Saturation Percent of the soil. pH is measured directly on the paste to determine the acidity of the soil while the extract obtained from the paste is used to measure soluble salts and conductivity.

The State of Nevada has specific requirements that must be met for NV-certified work, which are different from historical methods performed at ACZ. Communication with PMs is key when performing NV work.

4.0 SUMMARY

100 –300 g of **<250um (if mining and overburden) or <2000 (if agronomic) or 95% passing 150µm (if NV certified work)**, air-dried soil is mixed with sufficient Type 1 H_2O to saturate the soil, which is then mixed by hand into a wet paste just at saturation. The saturated soil paste is allowed to stand overnight or for at least 4 hours, or 1 hour for NV work. The pH and moisture percentage (Saturation Percent) of the paste is measured. The paste is filtered using vacuum filtration and special porcelain funnels. The resulting extract is analyzed for conductivity, major cations (Calcuim, Magnesium, Potassium and Sodium), and sometimes major anions such as Chloride, Nitrate and Sulfate. Additionally the Sodium Absorption Ratio (SAR) can be calculated.

5.0 REFERENCE

5.1. EPA Method 600/2-78-054, Section 3.2.2.

5.2 “Diagnosis and Improvement of Saline and Alkali Soils,” Method 2 - Saturated Soil Paste, pg. 84

5.3 “Diagnosis and Improvement of Saline and Alkali Soils,” Method 21a - pH Reading of Saturated Paste, pg. 102

5.4 “Diagnosis and Improvement of Saline and Alkali Soils,” Method 27a - Saturation Percentage from Oven Drying, pg. 107

5.5 Methods of Soil Analysis ASA No. 9, part 2-1982 method 25-9.2 Saturation Extract

5.6 Method SM2510B – for Electroconductivity

5.7 Nevada Modified Sobek Procedure, 2015 update

6.0 SAMPLE COLLECTION, HANDLING & PRESERVATION

6.1 Samples must be air dried, screened or crushed and pulverized to **<250um if mining and overburden, or dried and screened to <2000 if agronomic or 95% passing 150µm (if NV certified work)**.

6.2 **Table 6.1**: Hold Time Table for Saturated Paste Extractions

Parameters	Matrix	Hold Time
SP-PH, SP-EC	All Matrices	No Hold Time
SP-ORP		
SAT-PCNT		
Metals and Ions via Sat-Paste		180 Days from Collection Date to Analysis Date

6.3 Filtered extracts are kept in capped 15mL centrifuge tubes and are stored at room temperature.

7.0 APPARATUS & SUPPLIES

7.1. Plastic drinking cups, 12 oz. with lids
7.2 Stainless steel mixing spatula
7.3 pH meter
7.4 EC meter
7.4 Porcelain saturated paste funnel, 17 cm
7.5 Plastic mesh screens, 10 cm
7.6 Whatman 540 or 40 filters
7.7 Disposable aluminum weighing dishes
7.8 Vacuum filter station
7.9 Centrifuge tubes, plastic 15mL with caps
7.10 Centrifuge tube rack
7.11 Analytical balance capable of weighing to 0.01g, calibration verified daily (refer to SOPAD013).
7.12 Drying oven constant to 105°C

8.0 REAGENTS & STANDARDS

8.1. Type I H_2O. For Nevada certified work, Type I H2O is certified CO2-free.

8.2 Buffer 4 for pH LCSS. Manufacturer's storage and expiration apply. Stored in the Wetchem Manual department and in the soils digestion lab.

8.3 Nevada samples use NBM-1 for pH LCSS. Stored at room temperature. If supplied, manufacture's expiration date is observed; otherwise, material expires 5 years from receipt.

8.4 Ag/Ag Cl Reference Electrode filling Solution. Store at room temperature in the dark. Shelf life is two years from open date.

8.5 Fisher Scientific (or equivalent) pH Buffer solutions 2.0, 4.0, 7.0, 10.0, 12.0. Manufacturer's expiration dates apply. Stored on the soils digestion lab or in the Wetchem department.

8.6 Conductivity standards are purchased premade from a vendor; The standard and the LCS must be obtained from different sources. Manufacturer's expirations apply. Both, the calibration and LCS Conductivity standards are obtained from the Wet Chemistry Manual Department, and stored at room temperature in the Extraction Lab.

9.0 SAFETY

9.1 HAZARDS

All procedures within ACZ pose some safety hazards that may be avoided with attention to detail.

9.2 SAFETY TECHNIQUE

9.2.1 Safety glasses are required and the use of gloves and lab coat is strongly recommended. Shorts and open-toed shoes are not allowed in the lab.

9.2.2 Use care when pouring and pipetting reagents. Always add acid to water. Use the proper method when washing glassware.

9.2.3 Do not eator use tobacco products in the lab.

9.2.4 Wipe up ALL spills immediately. Implement the Emergency Response Plan if necessary.

9.2.5 Do not wear gloves or lab coat outside of the laboratory. Remove gloves before using a computer, telephone, etc.

9.2.6 Do not conduct "experiments" unrelated to the analysis.

9.3 PROTECTIVE EQUIPMENT

9.3.1 Use a fume hood when there is a potential for strong fumes.

9.3.2 A fire extinguisher is located in each analytical laboratory.

9.3.3 An emergency shower and eye wash station is located in the soils prep lab.

10.0 INTERFERENCES

10.1 Samples with high clay content require that water be slowly added to the sample and allowed to sit without mixing for several hours. This procedure allows the soil to absorb the water. Samples with high sand content require small additions of water to avoid waterlogging the sample. These samples may appear too dry, but upon settling, excess water will come to the surface.

11.0 PROCEDURES

11.1. Create a workgroup in LIMS, Soil Prep (20), "SP-G-SP," then create a workgroup in LIMS, Soil Analysis (25), "SA-G-SAT-PASTE." The analytical workgroup will copy the prep workgroup QC automatically. Modify the workgroups to include only those samples that are prepped and ready for analysis. Assess whether the samples are mining and overburden, or agronomic soil samples. Samples that are mining and overburden are air dried, crushed and pulverized to <250 um. Samples that are agronomic are air dried and sieved to <2000µm, but NOT CRUSHED AND PULVERIZED. Samples from Nevada must be 95% passing 150µm (if NV certified work). If the samples are in for cations they will be associated with the prep workgroup.

11.2 Gather and organize the samples by lab number and mark one plastic cup per sample with a corresponding lab number.

11.3 Fill each 12 oz plastic cup approximately 1/2 to 3/4 full with sample. If pH is the only product being analyzed, a smaller sample can be mixed into a centerfuge tube.

NOTE: For each client sample, verify that the sample Log-in number on the workgroup matches the sample number on the bottle. This ensures that samples are not inadvertently switched.

11.4 For Nevada ONLY: Steps 11.5 through 11.9 are skipped. Instead, water is added at a 1:1 ratio. The samples are allowed to sit for about 1 hour, and the pH of the slurry is recorded to 1 decimal place. This procedure is contrary to historical methods and data produced by ACZ, and the data generated should not be compared to data produced by the traditional saturated paste method.

11.5 Add water (approximately 1/4 to 1/2 the volume of soil sample) to each cup and sample. The volume of water needed to saturate the soil depends on the type of soil. Add water in small increments to sandy soils

11.6 Mix the samples with a clean stainless steel spatula when they have fully absorbed the water. Clean the spatula with water and wipe with a clean paper towel between samples.

11.7 Slowly add more water while mixing, to the point where the paste glistens as it reflects light, flows slightly when the container is tilted and slides off the spatula when tapped. After mixing, tap the plastic container on the bench to settle the paste. If the water content is correct, there should be no free standing water. If excess water has been added to the sample, add small amounts of additional soil while mixing to absorb the excess water. You are trying to achieve the point at which the soil becomes saturated.

NOTE: Samples with high clay content will not slide off the spatula and may require up to 100% of the soil weight in water to fully saturate it. These samples tend to ball-up during the mixing process. Excess water will come to the surface upon settling for sandy soils. Saturation percents will follow these guidelines: sandy soils - 10 to 30%, loamy soils - 30 to 60%, clay-like soils - greater than 60%.

11.8 If the samples are high in clay or organic matter, check the samples periodically throughout the day. If the paste has stiffened or lost its glisten, add more water and remix. If free water has collected on the surface, add additional air-dry sample to absorb the free water and remix.

11.9 Cover the samples and let them sit over night.

11.10 Check the samples the next morning for saturation and repeat step 11.7 if needed. If additional mixing is required the samples must set for a minimum of 4 hours.

11.11 Determining the Saturation Percent

11.11.1 Label one aluminum weigh dish for each sample in the workgroup. Create one PBS per 20 samples and record on benchsheet. PBS data will not be entered into LIMS.

11.11.2 Zero the balance, weigh and record the pan weight on the workgroup (dept 25).

11.11.3 Mix the paste until it is homogenized. Transfer 10 to 20g of paste into the dish, weigh and record.

11.11.4 Place the samples on a drying pan to keep them in order.

11.11.5 Place the pan and samples in the drying oven at 105°C overnight.

11.11.6 Remove the samples from the oven and allow them to reach room temperature.

11.11.7 Weigh and record the samples. Do not discard them until the data has been entered and checked for obvious errors.

11.12 Determining the pH of the Saturated Paste

11.12.1 Refer to SOPSO010 for proper operation and calibration of the pH meter.

11.12.2 The pH of the paste is measured directly by slowly inserting the pH probe into the mixed paste and wait for it to stabilize. While you are waiting for it to stabilize, mix the next sample to be analyzed. Care should be taken not to break the fragile pH probe bulb when inserting it into sandy soils.

11.12.3 Record the pH value to 0.01 pH units.

11.13 Filtration of the Saturated Pastes

11.13.1 Label a 15mL centrifuge tube (LIMS #, x-sp and date) for each of the samples and place them in order in a labeled tube rack (WG#, LIMS #, x-sp and date).

11.13.2 Rinse the saturated paste porcelain funnel and the funnel screen with DI and then Type I H_2O. Place them on clean paper towel to drain.

11.13.3 Place the clean funnel into the top of the vacuum filter station. Place a plastic cup inside the filter station to collect any excess water.

11.13.4 Place the funnel screen onto the funnel.

NOTE: Pre-saturate a Whatman 540 (or 40) filter by placing it on the porcelain funnel and screen and completely wetting with Type 1 water.

11.13.5 Turn on the vacuum pump to seat the filter paper. When the excess water from the saturated filter is pulled into the plastic cup flask, turn off the pump and remove the cup. Replace the cup with a labeled 15mL centrifuge tube. Turn the pump on again.

11.13.6 Slowly pour and spread the paste over the entire area of the filter paper.

11.13.7 Continue filtration until the paste is dry and no more extract is dripping into the 15mL centrifuge tube

Minimum Extract volumes:

EC	4 mL
Cations	8 mL

11.13.8 Remove the centrifuge tube, cap it and place it in the rack

11.13.9 Repeat cleaning steps 11.13.2 –11.13.5. Be careful not to throw away the funnel screen with the spent paste. Avoid rinsing the soil down through the center of the funnel.

11.13.10 Continue until the workgroup is complete. The entire workgroup must be filtered on the same day.

11.14 Determine the conductivity of the Saturated Paste

11.14.1 Calibrate the EC meter

11.14.2 Turn battery operated meter on

11.14.3 Label a 15mL centrifuge tube “STD” and pour about 4mL of the Traceable conductivity Calibration Standard in it.

11.14.4 Label another 15mL centrifuge tube “LCS” and pour about 4mL of the check standard 0.01 M KCl.

11.14.5 Remove the guard from the conductivity probe and submerge it in the Conductivity Standard. Move the probe up and down to remove any air bubbles that may interfere with the probe readings.

11.14.6 Press the CHECK key, “CHK” will appear on the top right corner of the display.

11.14.7 Press and hold the ENTER key for 10 seconds, “0”zero will be displayed to the far left of the display to indicate that all calibration data has been cleared.

11.14.8 Press the appropriate arrow to set the True Value uS/cm from the standard bottle.

11.14.9 Press enter to set and record, in the logbook, the observed value after setting. Record the temperature.

11.14.10 Rinse the probe and pat dry.

11.14.11 Submerge the probe in the check standard. Move the probe up and down to remove any air bubbles that may interfere with the probe readings.

11.14.12 Let the reading stabilize and record the observed value in the logbook. Record the temperature.

11.14.13 Calculate the percent recovery and record. Recovery needs to be 90% - 110%, if it is not, remake the check standard, change the battery, check the expiration date on the standard, and check your observed values with the Wet Chem EC meter.

11.14.14 Remove one sample cap at a time so sample does not spill from one sample to the next.

11.14.15 Slowly insert the EC probe into the sample just enough so the probe electrode is submerged. Swirl the probe to remove any air pockets around the probe.

11.14.16 Allow the reading to stabilize and record the reading as it appears in uS/cm or mS/cm on your benchsheet. Final reporting of data requires the units to be entered mmhos/cm. Reference section §13.0 to make sure you are converting from µS/cmto mmhos/cm correctly. Record the temperature.

11.14.17 Between EC measurements, thoroughly rinse the electrode with Type I H_2O and pat dry. Carefully shake out any water that remains inside the conductivity cell.

11.14.18 Continue until workgroup is complete.

11.15 Enter the data and upload both workgroups to LIMS. The column for max. particle size is used to differentiate between agronomic samples (max particle size 2000 um) and mining and overburden samples (max. particle size 250um) and is reported to clients. Make sure this workgroup # is on the sample rack. The workgroup for dept. 25 needs one copy for reporting.

11.16 AREV data in LIMS and turn in to your supervisor for SREV.

11.17 . Place samples in for cations in the metals storage room.

12.0 QUALITY CONTROL

12.1. One duplicate (DUP) is required for each workgroup of 20 or less client samples. Choose duplicate samples which have sufficient volume and are logged in for the most comprehensive set of analysis.

12.2 Saturation percent requires a blank to check for proper oven control, however there is not a row for it so hand write the data above the appropriate columns.

12.3 Calculate the DUP RPD for pH, EC and Saturation Percent:

$$[(X_1 - X_2)] / [(X_1 + X_2) / 2] \times 100$$

12.4. The analysis is acceptable if the DUP RPD $\leq$ 20 (provided sample value > 10x MDL).

12.5. The analyst will request re-prep of samples if QC is outside of the acceptance limits.

12.6. An LCS is required for saturated paste pH. ACZ currently uses buffer 4 for non Nevada samples and NBM-1 for Nevada samples. Hi/low limits have been established at +/-3X the standard deviation found in the NBM-1 Certificate of Analysis.

13.0 DATA CALCULATIONS, DATA REPORTING & ARCHIVING

13.1

$$\text{Saturation Percent} = \frac{(\text{Wet sample \& Cruc. wt.gm}) - (\text{Dry sample \& Cruc. wt. gm})}{(\text{Dry sample \& Cruc. wt. Gm}) - (\text{Cruc. Wt. gm})} \times 100$$

Saturation percent should correlate with the Texture analyses of the samples. Conductivity, when multiplied by 10 to 13, should approximate the sum of the major cations.

13.2 µS/cm divided by 1000 = mmhos/cm

13.3 mS/cm = mmhos/cm

13.4 SAR = (Na) / (the square root of Ca + Mg / 2) where Na, Ca and Mg refer to the concentrations of designated cations expressed in milliequivalents per liter (meq/L).

13.5 pH MDL is 0.1 units

13.6 EC MDL (precision) is 1µS/cm

13.7 Saturation percent MDL is 0.01 %

13.8 Saturation percents will follow these guidelines:

sandy soils - 10 to 30% loamy soils - 30 to 60% clay-like soils - greater than 60%.

14.0 METHOD PERFORMANCE/DETECTION LIMITS

14.1 There is no Method Detection Level (MDL) study required for this procedure.

14.2 Demonstration of Capability (DOC): A successful Initial DOC must be completed and approved by the QA/QC department for each analyst prior to independent generation of client data. A Continuing DOC is required annually.

Prepare & analyze 4 individual aliquots of a reference material (LCSS) certified for saturated paste pH. Currently, buffer 4 is the certified standard for saturated paste pH. Enter all data on form FRMAD023 in Qualtrax. Print the completed form, sign it and submit it with all raw data and any other supporting documentation required for an environmental sample to the department supervisor for review. Refer to SOPAD001 for more details.

15.0 DOCUMENTATION

15.1 Record the appropriate information for all calibrations the appropriate logbook.

15.2 Make sure the following information is included on the Workgroup:

- Analysis date, start time and completion time.
- Analytical method used.
- Analyst’s initials.
- Calculated QC data results.
- Any remarks about analysis or samples.
- Data review/checklist form.
- Standard/reagent prep sheet, if applicable.
- *ANY OTHER PERTINENT INFORMATION*

16.0 WASTE MANAGEMENT/POLLUTION PREVENTION

16.1 Refer to ACZ’s Waste Management Plan for appropriate disposal details for this method.

16.2 Spent saturated paste is disposed of in the garbage.

17.0 DEFINITIONS

17.1 Prep Blank (PBS) –A reagent blank carried through the entire preparation scheme. It is treated exactly as a sample, including exposure to all glassware, equipment, and reagents, and it confirms the absence of contamination in the prep stages.

17.2 Duplicate (DUP) –A second aliquot of a client sample prepared and analyzed like all other client samples in the same workgroup. The DUP demonstrates the precision of the method.

17.3 RPD (Relative Percent Difference) –The difference between two replicates (a sample and its duplicate) divided by the average of those two replicates times 100.

17.4 MDL (Method Detection Limit) –The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero.

18.0 TABLES & DIAGRAMS

18.1 **Table 6.1**: Hold Time Table for Saturated Paste Extractions

19.0 CORRECTIVE ACTION

19.1 For QC samples that do not meet the method acceptance criteria, refer to §12. For retests that will occur past the method hold time, check first with the department supervisor to determine if the reanalysis should be conducted.

19.2 For any SOP/method deviation fill out section 1 of a corrective action report (FRMQA001). If necessary, the department supervisor and/or project manager may provide additional information in the appropriate sections; however, QA does not need to close a minor corrective action. Attach a copy of the minor corrective action report to all workgroups affected. A minor corrective action is for documentation purposes – any SOP or method deviation may be noted on the data review checklist or on the workgroup bench sheet in lieu of using FRMQA001.

19.3 For any system failure a major corrective must be opened and the problem investigated. Consult the QA department to open a major corrective action. The corrective action will be assigned a unique tracking number by the QA Officer (or designee) and will be closed by the QA Officer (or designee) once the failure has been resolved. .